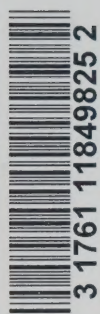


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Royal Commission on Matters of Health  
and Safety Arising from the Use of  
Asbestos in Ontario

MEASUREMENT OF ASBESTOS FIBRE  
CONCENTRATIONS IN WORKPLACE ATMOSPHERES

A Study Prepared By:

Dr. Eric J. Chatfield  
Ontario Research Foundation

**Study Series**





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for

**The Royal Commission on Matters of Health and Safety**

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\* \* \* \* \*

This study was commissioned by the Royal Commission on Asbestos, but the views expressed herein are those of the author and do not necessarily reflect the views of the members of the Commission or its staff.


November 1982





PREFACE

This document contains a review of the current methods for monitoring of asbestos fibre concentrations in workplace atmospheres for determination of compliance with legislated control limits. Every attempt has been made to incorporate the most recent published work in this area; however, in many relevant publications, insufficient detail has been provided to allow useful direct comparisons with other observations. Much of the work discussed has not yet been formally published, and originates in some cases from internal reports.



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## 1. INTRODUCTION

Airborne asbestos is monitored in the workplace atmosphere in order to establish compliance with regulations, to check the performance of dust control equipment, and also to provide exposure data for epidemiological studies. For each of these purposes, specialized sampling or measurement strategies may be defined.

For assessment of the exposure of workers to airborne asbestos fibres, it is important that the measurement should be representative of the airborne dust at the work station. Thus, the number and locations of the samples taken should be selected so that variations of concentration with both time and position can be determined. Many workers perform a series of tasks during the day's work, and so may be exposed to a wide range of fibre concentrations. Under these circumstances, the use of fixed-position air samplers at each work location would not provide a representative picture of worker exposure.<sup>1</sup> In the majority of cases, a portable sampler carried by the worker, such that air from the nose and mouth region is sampled, offers the best method for obtaining samples which are representative of the individual worker's exposure. The portable sampler, referred to as a personal sampler, is light, battery-operated, and is only a minor inconvenience to the worker when used for occasional measurements.

Measurement of the amount of asbestos collected by the sampler can be specified in terms of either mass, or the number of fibres. It is important that the parameter measured should correspond to the property thought to be responsible for the biological effects. Several problems are encountered when attempting to relate a mass measurement to worker exposure. The measurement of total mass is inappropriate for estimation of worker exposure when the majority of the total dust is not asbestos, for example, in the asbestos-cement industry. Even if the total dust measurement is refined to include a specific determination of the proportion which is asbestos, there is still a difficulty in that the larger fibres



contribute to the mass measurement, but these large fibres would not penetrate to the lung alveoli. Size selection instruments, which allow separation of the respirable particles by their aerodynamic properties,<sup>2</sup> could possibly be used to reject the non-respirable material, but the sampling characteristics of such instruments are not satisfactory for use with fibrous dusts.<sup>3</sup>

Legislation in most countries is now based on measurement of the numerical concentration of airborne fibres using specified methods of microscopical examination. Among the developed countries, Germany and the USSR are the only ones to retain a standard based on measurement of mass, although in Germany numerical standards are now used.<sup>4</sup>

Several versions of a method for counting fibres on filters have been developed,<sup>5-9</sup> all of which are based on phase contrast optical microscopy. The results obtained by these methods, however, are affected by subjective factors, and those inter-laboratory results which are available display considerable variability. It has been found that groups of fibre counters can be brought into accord with each other by a continuous programme of sample exchanges, and this appears to be the only realistic way of using the method.

Clearly, one way of removing the subjective factors is to automate the fibre counting process. Automatic fibre counting instruments, applied to both optical and scanning electron microscopy, are under investigation.<sup>10-12</sup> However, these procedures are at an intermediate stage of development and have yet to achieve general acceptance on considerations of both cost and performance.

Automatic fibre counting instruments which count fibres while still suspended in air have been developed.<sup>13</sup> Unfortunately, these instruments are unsuitable for personal sampling, and they also present some calibration difficulties. Another measurement method, based on magnetic alignment combined with scattered light measurements, has resulted in production of an instrument for measurement of fibre



concentrations on filters.<sup>14</sup> This instrument, however, yields results which require individual calibration work for each size distribution encountered, and consequently interpretation is difficult. Nevertheless, the basic technique is capable of considerable development, and has the advantage that it produces a physical measurement which is not subject to the statistical limitations on the precision imposed by counting of fibres on only a small fraction of the total sample. Moreover, unlike the situation for manual fibre counting, operator subjectivity does not affect the measurement.

Particle detection and mass measurement instruments such as the Tyndallometer<sup>15</sup>, the respirable dust monitor<sup>16</sup>, and the ambient particulate monitor<sup>17</sup> are valuable in a general programme of engineering control. These instruments permit early warnings to be obtained of deviations from normal performance, and also allow the effectiveness of improvements to be monitored more completely than could be achieved by manual fibre counting methods. However, they do not currently permit discrimination of fibrous from non-fibrous dusts, and therefore cannot be considered as suitable for compliance determination.

This report considers the requirements for measurement, and how the current analytical methods meet these requirements. The impact of future developments on the measurement methods is also considered.

## 2. MEMBRANE FILTER METHODS

Personal air samples are collected on cellulose ester membrane filters of pore size 0.8 micrometre ( $\mu\text{m}$ ) or 1.2  $\mu\text{m}$ . The filter is then cleared (made transparent), and the number of fibres which have prescribed dimensions are counted over a known area. The mean fibre concentration over the period during which the sample was collected can then be calculated.

### 2.1 Fibre Definition

The original membrane filter method<sup>5</sup> was designed to measure fibres thought to be responsible for lung damage and, in particular, asbestosis. At

the time it was thought that only the longer fibres were of concern, and it was decided to measure only those longer than 5  $\mu\text{m}$  and shorter than 100  $\mu\text{m}$ . The upper limit is now not normally applied, but usually the proportion of fibres longer than 100  $\mu\text{m}$  is small and the difference between the results is insignificant. Rather arbitrarily, a length:diameter ratio of at least 3:1 was selected as the definition of a fibre. A fibre must be able to penetrate to the lung in order for it to cause lung disease. Accordingly, a further limitation on the fibre definition can be made on the basis of its aerodynamic properties. Although lung models which express the retention of spherical particles have been published,<sup>18-19</sup> their application to fibrous particulate is uncertain. Timbrell<sup>20</sup> showed that fibres fall through air at rates which are dependent on their diameters, and almost completely independent of their lengths. Timbrell's results indicated that asbestos fibres behave as if they had aerodynamic diameters equal to almost three times their physical diameters. On this basis it can be concluded that fibres with diameters exceeding about 3.5  $\mu\text{m}$  are not respirable; consequently, some versions of the fibre definition incorporate a maximum diameter of 3  $\mu\text{m}$ , although 5  $\mu\text{m}$  is sometimes used. In summary, the fibre definition dimensional criteria are:

- (a) an aspect ratio equal to or greater than 3:1;
- (b) a length greater than 5  $\mu\text{m}$ ;
- (c) a diameter less than 3  $\mu\text{m}$  (optional).

A further requirement is that the fibre should be visible at a magnification of 500 in phase contrast illumination. Asbestos fibres with diameters less than about 0.2  $\mu\text{m}$  are not visible in the phase contrast optical microscope, regardless of their lengths.

It must be recognized that the membrane filter measurement provides only an index of the exposure. Moreover, the fibre definition is based on dimensions only, and no attempt is made to apply additional constraints on

the basis of morphology or "appearance", although many mineral species other than asbestos have similar morphology, e.g., organic fibres and gypsum.

In attempts to improve the reproducibility of optical fibre counting, changes in the fibre definition have been considered. It has been reported that a change to a minimum aspect ratio of 5:1 would significantly improve reproducibility.<sup>21</sup>

## 2.2 Published Fibre Counting Methods

Although the membrane filter method is often thought of as a single technique, there are a number of variations in use, and one of these is currently under consideration by the International Organization for Standardization (ISO), as a Draft International Standard (DIS).<sup>9</sup>

### 2.2.1 The Asbestosis Research Council (ARC) Method<sup>5</sup>

In this method the dust particles are fixed on the membrane filter surface using a solution of polymethyl methacrylate in chloroform, and the filter is then cleared by placing it on a drop of glycerol triacetate (Triacetin).

### 2.2.2 The U.S. Public Health Service, National Institute for Occupational Safety and Health (USPHS/NIOSH) Method<sup>6</sup>

The mounting and clearing medium used in this method is a mixture of dimethyl phthalate and diethyl oxalate, with the addition of cellulose ester filter material to increase its viscosity. Depending on the area of the microscope graticule selected ( $0.003 \text{ mm}^2$  or  $0.006 \text{ mm}^2$ ), the recommended minimum fibre loading is either 33 fibres/ $\text{mm}^2$  or 17 fibres/ $\text{mm}^2$ . The corresponding recommended maximum fibre loading is either 1667 fibres/ $\text{mm}^2$  or 833 fibres/ $\text{mm}^2$ , with no additional restrictions for different types of samples.

#### 2.2.3 The Ontario Ministry of Labour (MOL) Method<sup>7</sup>

This method is similar to that published by the USPHS/NIOSH, with some modifications, omissions and additions.

#### 2.2.4 The Asbestos International Association (AIA) Method<sup>8</sup>

The AIA Reference Method, published in 1979, uses the filter mounting and clearing technique originally introduced by the Australian Department of Health.<sup>22</sup> The filter structure is collapsed by exposure to acetone vapour, after which the filter is cleared and mounted, using Triacetin. The AIA method takes into account most of the instrumental and other factors which have been shown to produce variability, and the intention in the method is to standardize all parameters so far as is possible. Precise fibre counting rules are also incorporated. The recommended minimum and maximum fibre loadings are 50 fibres/mm<sup>2</sup> and 637 fibres/mm<sup>2</sup> (5 fibres/field of view) respectively. It is stated that the maximum value should be reduced to 127 fibres/mm<sup>2</sup> (1 fibre/field of view) for samples of mixed dusts or when agglomerates are present, but this can be increased to 1273 fibres/mm<sup>2</sup> (10 fibres/field of view) if only fibres are present. It is recommended that filters which have average loadings exceeding 1273 fibres/mm<sup>2</sup> (10 fibres/field of view) should be rejected.

#### 2.2.5 The International Organization for Standardization Draft International Standard (ISO/DIS) Method

This method, which is still under development, is based on the AIA method. A working group of the International Organization for Standardization (ISO) is revising the method to incorporate the best available procedures. In association with this working group, a bi-lateral committee of experts from Canada and the Commission of European Communities (CEC) has investigated three versions of fibre counting rules to determine their effect on the variability of the final result.<sup>23</sup>



#### 2.2.6 Other Versions of the Membrane Filter Method

Variants of the basic membrane filter method have usually involved either the filter clearing and mounting procedure, or the fibre counting rules. LeGuen and Galvin<sup>24</sup> have described a method in which the membrane filter is cleared using a mixture of dimethyl formamide, acetic acid and water. After drying, a drop of a thermo-setting resin, Euparal, is added, followed by a cover glass. Euparal can also be used as a replacement for the Triacetin as used in the AIA method. LeGuen and co-workers<sup>25</sup> have also examined the possibility of exposing the fibres embedded in a membrane filter after the acetone vapour treatment. Their method involves exposing the filter to an oxygen plasma, which etches the filter surface, leaving the fibres exposed but still attached. The refractive index of the immersion medium can then be selected by the microscopist to yield increased fibre visibility. It is reported<sup>26</sup> that studies conducted in the United Kingdom showed that samples mounted by the acetone-Triacetin technique of the AIA method displayed some fibre movement. Accordingly, the Central Reference Laboratory (CRL) of the U.K. has recommended the use of an acetone-Euparal method for preparation of filters as permanent reference slides. The CRL also employs an alternative system of fibre counting rules.<sup>27</sup>

### 2.3 Comparison of the Published Membrane Filter Methods

It would be neither practical nor useful to discuss the intricate detail of each method, but there are some significant differences which must be considered in relation to the Ontario MOL method.

#### 2.3.1 Filter Mounting Technique

The work by LeGuen and co-workers<sup>25</sup> indicated that the detection limit for chrysotile fibres on membrane filters occurred at

a diameter of about 0.20 - 0.25  $\mu\text{m}$ . Rooker et al<sup>28</sup> reported that, provided the difference between the refractive indices of the fibre and its immersion medium exceeded 0.06, fibres with diameters down to 0.15  $\mu\text{m}$  should be detectable. Teichert<sup>29</sup> found that, using the acetone-Euparal mounting technique, the fibre counts for chrysotile were only 70% of those obtained by the acetone-Triacetin method. Furthermore, using test slides it was found that the resolution limit was lower for the acetone-Triacetin method than for both the acetone-Euparal method and the USPHS/NIOSH mounting medium. Smaller differences between the fibre counts obtained when using the acetone-Triacetin, the acetone-Euparal and the dimethyl formamide-Euparal techniques were found by Ogden.<sup>30</sup> However, Teichert<sup>29</sup> commented that divergences between the results for the three mounting media were sometimes insignificant, but only when predominantly thick fibres were present on the original filters; thus differences in the diameter distributions may account for the difference between the two studies.

Both of the Euparal techniques and the AIA acetone-Triacetin method produce slides which are permanent and available for later evaluation. The USPHS/NIOSH and the Ontario MOL methods produce slides which must be counted within about 2 days and which are useless thereafter.

It is recommended that the MOL method be changed to incorporate the acetone-Triacetin preparation step. It is also recommended that the counted and labelled slides be stored, along with the remaining filter segments, as permanent records of the measurements.

### 2.3.2 Specification of Microscope Alignment and Performance

It has been demonstrated by Beckett and Attfield<sup>31</sup> that incorrect alignment of the phase contrast microscope can result in significant reductions in fibre counts. Teichert<sup>29</sup> has also shown that the resolution limit can be seriously degraded by a number of factors, including maladjustment of the phase rings. The AIA method and the proposed ISO/DIS method both clearly specify the use of test slides to ensure adequate microscope resolution. The other published methods do not. It is recommended that the MOL method be modified to incorporate tests of the microscope resolution.

### 2.3.3 Graticule Selection

The purpose of the eyepiece graticule in a microscope is to provide reference dimensions for measurement of the particles, and precise delineation of a known area of the filter. Beckett and Attfield<sup>31</sup> observed that the use of a graticule which defined a counting field of view of only 5 - 20% of the total field of view resulted in higher fibre counts than those obtained using full-field counting. Their study yielded the unexpected conclusion that chrysotile samples assessed by counting fibres only within the graticule area produced results three times higher than those obtained by counting fibres over the whole field of view. The effect was somewhat lower for amosite. When additional time was spent in rigorous evaluation of each field of view, this difference was reduced significantly.

Four types of graticule have generally been used for counting of asbestos fibres:

- (a) the Patterson Globe and Circle;
- (b) British Standard 3625;
- (c) the Porton; and
- (d) the Walton/Beckett.

Only the Walton/Beckett graticule<sup>32</sup> has been designed specifically for fibre counting. It is the only graticule which does not demand visual interpolation by the operator in order to determine whether fibres exceed the critical dimensions. The Walton/Beckett graticule also defines a standard area of the sample, and therefore the component of the variability associated with the "graticule effect" can be eliminated.

In both the AIA and ISO/DIS methods this graticule has been adopted as standard. It is recommended that the MOL method should specify the Walton/Beckett graticule exclusively.

#### 2.3.4 Quality Control Procedures

The latest (1979) revision of the USPHS/NIOSH method and the MOL method incorporate criteria for re-counting of samples. The USPHS/NIOSH method recommends that 1 in 10 samples should be counted *blind* by either the same operator or a second operator, although in the same method it is specified that blank filters be labelled as such. Rejection criteria are also specified, but in view of controversy surrounding the actual values of the coefficient of variation (CV) of the method (discussed in Section 2.4.3), these should be viewed with some caution. Nevertheless, the USPHS/NIOSH method recognizes that counts by a second operator may be introduced as a quality control measure. In contrast, the MOL method specifies re-counting of "one sample in every twenty or one sample per batch, whichever is less." This wording is ambiguous, but could represent a substantial reduction of the quality assurance level. Moreover, re-counting *by the same operator* is the only quality assurance measure specified in the MOL method. Limitation of the re-count requirement to the same operator ignores the most significant variability factor. The inter-laboratory, inter-operator variability is very large (discussed in Section 2.4.3), and it is clear that blind re-counts of samples by other operators should be incorporated in the determination of compliance, and should preferably be performed by an independent laboratory.



### 2.3.5 Fibre Counting Criteria

The membrane filter method is an analytical technique which differs from measurements of many other hazardous agents, in that changes of fibre definition or fibre counting criteria produce not only changes in precision, but also changes in the reported fibre concentration. Any such change is also liable to alter the *relative* values obtained in specific types of asbestos use or industry. Although the AIA counting criteria represent the most detailed published tabulation for counting of both single fibres and more complex assemblies, another system is in use in the United Kingdom and is thought to have the advantage of simplicity.

The United Kingdom CRL fibre counting criteria<sup>27</sup> are an attempt to reduce the effects of operator subjectivity, and differ from the AIA criteria principally in the treatment of split fibres, and fibres grouped with other fibres or particles. A particularly contentious feature is the counting of fibre aggregates which are too complex for evaluation of the number of component fibres. Whereas the AIA criteria specify that such features should be counted as zero, the CRL rules assign an arbitrary value of 8 to each. Where much of the airborne asbestos occurs as aggregates, as in the asbestos-cement industry, use of such a rule may drastically and arbitrarily increase the reported fibre concentrations. In other industries the effect may be less evident. So far as can be ascertained, the effects of using the CRL criteria have not been evaluated on mining or milling air samples. Nevertheless, there are indications from the study commissioned by Canada and the CEC<sup>23</sup> that use of a modified version of the CRL fibre counting criteria may lead to improvements in the inter-laboratory variability. It is also thought that change of the aspect ratio definition to  $\geq 5:1$  would yield an improvement. However, pending a full interpretation of the effects of these modifications on samples from specific types of industry, the AIA criteria represent

the most complete system of rules which have currently attained international acceptance. It is recommended that these should be adopted in the Ontario MOL method, until the criteria for the ISO/DIS method have been finalized.

#### 2.3.6 Membrane Filter Method: Summary of Recommendations

The many advantages of the published AIA method lead to the conclusion that the principal features of this method should be adopted by Ontario. The acetone-Triacetin method of mounting the filter provides a permanent sample which can be used as archival material for future study as required. Furthermore, this mounting method provides the best fibre visibility for chrysotile, which is the most frequently encountered variety and is also of most importance in Canada. It has been demonstrated that the acetone-Triacetin method leads to only slightly higher average fibre counts than those produced by the USPHS/NIOSH method.

The eyepiece design of Walton/Beckett should be adopted in the method used by Ontario. It has been shown that the subjective effect is the most significant component of the errors in the membrane filter method, and any such improvement which reduces the latitude for subjective decisions should be adopted.

The fibre counting criteria published in the AIA method should be adopted for the Ontario MOL method as an interim measure, since they represent a current level of international consensus. The CRL criteria can be considered to discriminate unduly against the asbestos-cement industry, and have not been evaluated on mining or milling samples. When agreement is reached on the criteria to be adopted for the ISO/DIS method, these should also be adopted by Ontario.

The other major recommendation concerns quality assurance. In view of the demonstrated high inter-laboratory, inter-operator variability of the fibre counting method, it is clear that the quality assurance procedures incorporated in the MOL method are insufficient. Moreover, in both Ontario and the rest of Canada, no control is currently exercised over the quality assurance of membrane filter fibre counting by any laboratory, whether government, independent or commercial. Inter-laboratory variability and the requirement for greater control will be addressed further in Section 2.4.3. It is, however, strongly recommended that the MOL method be modified so that:

- (a) a more rigid system of quality assurance, including blank and control samples, blind re-counting and external controls, be incorporated as a mandatory requirement in the MOL method; and
- (b) an independent (not government or industry) laboratory be charged with the responsibility of operating a central reference system against which government, industry and commercial laboratories are required to maintain proficiency.

## 2.4 Limitations of the Membrane Filter Method

### 2.4.1 Measurement Errors and Interpretation

Measurement of airborne asbestos fibre concentrations is subject to a number of errors, both systematic and random. These can be separated into errors associated with sampling, and those which are incurred as a result of the method of analysis. The systematic errors which occur during sampling include those in flowrate determination, sampling period and the less controllable features such as non-representative sampling and sample contamination. The flowrate

and airborne fibre concentration may also vary randomly during the period of sampling. Errors introduced during analysis of the filter sample include those in determination of the effective filtration area and the analysis area, and those introduced by variability in filter mounting. The major errors in the analysis, however, appear to be associated with subjective effects such as the visual acuity and state of fatigue of the observer, physical factors such as the optical imperfections of both the sample and the microscope, and statistical effects which are a consequence of the distribution of fibres on the filter.

It is important to recognize that the membrane filter method does not allow an absolute value for the airborne fibre concentration to be obtained, and consequently all discussions relate to precision and "counting level." It is a relatively crude method by which only the larger diameter fibres are monitored, but if it is assumed that the systematic errors can be kept constant, the method can be used to compare measured values with an established value which is considered acceptable.

Studies have been made into the relationship of the membrane filter fibre count and actual chrysotile fibre concentrations determined by transmission electron microscopy (TEM) methods.<sup>21</sup> For samples with fibre loadings significantly higher than those recommended in the AIA membrane filter method,<sup>8</sup> the number of fibres longer than 5  $\mu\text{m}$  reported by the TEM measurement may be up to 100 times higher than the membrane filter values. The total number of fibres in all size ranges may be as much as 1000 times higher than those reported by the membrane filter method. The ratio between the values reported by the TEM and the membrane filter methods is significantly lower when the filters are loaded to concentrations within the AIA acceptable range.



However, although a relationship between the membrane filter methods and TEM fibre counts can be seen for chrysotile by itself, similar parallel results determined from samples obtained in the asbestos-cement industry have shown no correlation with each other.<sup>33</sup> It is therefore clear that when chrysotile is in association with other common industrial materials, the membrane filter method may yield values which cannot be compared with those obtained where chrysotile is manipulated by itself. More work is required on this topic in order to determine the extent of the problem. The existing data certainly indicate that membrane filter results in some industries may not reflect even the true *variations* of fibre concentration, and consequently that application of a single control limit to all industries may be questionable.

Since the visibility of an asbestos fibre in the phase contrast microscope is controlled by a combination of its diameter and the difference between the refractive indices of the fibre and the immersion medium, results obtained for chrysotile cannot be assumed valid for amphibole asbestos fibres. Additional studies are therefore required in order to determine the relationship between fibre levels reported by the membrane filter method and the actual airborne concentrations when amosite and crocidolite are present, either alone or mixed with chrysotile.

Because of intra- and inter-laboratory variability, it is even difficult to assess the repeatability, or precision, of the method. The systematic errors associated with measurements made by particular individuals take on a random appearance when sample analyses are conducted by numerous individuals in various states of fatigue using different microscopes. For the analyses to be useful in enforcement of control limits, it is important that the precision of a single measurement should be known. Unfortunately, no complete

study has yet been made which would represent the performance of the best available techniques. The USPHS/NIOSH technique and the modification of it published in 1982 by the Ontario Ministry of Labour do not specify some of the variables which have been found to be important in elimination of sources of systematic errors.<sup>34</sup> Nevertheless, in North America the basic USPHS/NIOSH technique is still the one specified by most regulatory bodies and the errors associated with this method are therefore the ones requiring assessment as to their compatibility with control limits.

#### 2.4.2 Fundamental Limitations of the Fibre Counting Technique

If it is at first assumed that the filter used for air sampling has collected a representative sample of the airborne dust cloud, and that all fibres of the specified dimensions have been reliably and reproducibly detected, the distribution of fibres on the filter defines the ultimate precision with which a fibre count can be obtained. In the absence of fibre-to-fibre interactions, the deposit of particles on the air sampling filter will be according to a Poisson distribution. Fibres are counted on only a small area of the filter, and on the basis of the count an estimate of the total filter loading must be calculated. In order to do this, the distribution of fibres on the filter must be known or assumed. The assumption that the fibres are deposited on the filter according to a Poisson distribution produces the narrowest range of probable error in estimating the mean fibre count; other random effects such as fibre-to-fibre interaction cause a broadening of the range within which the mean value can be stated for a specific degree of confidence.<sup>35</sup> Even if the actual distribution of fibres on the filter is Poissonian, characteristics of the individual operator and the microscope, together with the fibre counting criteria adopted, may cause the results reported to be non-Poissonian. For all practical purposes it cannot be assumed that the distribution is Poissonian. Since it is not standard practice in the USPHS/NIOSH method to record the fibre counts for individual fields of

view, little data exist as to the actual situation experienced in large numbers of routine industrial fibre counts.

Since adequate data could not be found in the literature, a study was undertaken in order to determine the extent to which deviations from the Poisson distribution occur. Additional records were made of the fibre counts on individual fields of view during the routine examination by a single operator of 91 air filters, collected in several different industrial locations. The results of the study are shown in Table 1. It was found that for filter loadings above about 170 fibres/mm<sup>2</sup>, the fibre distribution may depart significantly from that predicted by the Poisson distribution. Below this filter loading, the fibre counting data did not indicate a significant divergence from Poisson behaviour. However, for fibre loadings within the acceptable range for measurement, the possibility that the deposit of fibres on the filter is not Poissonian cannot be discounted. In Table 1, it can be seen that the proportion of non-Poissonian deposits was 40% for filter loadings within the range 170 - 655 fibres/mm<sup>2</sup>, which is still within the acceptable range of filter loadings specified by the AIA method. These results are in general agreement with those of a French study,<sup>36</sup> in which it was found that 211 out of 810 fibre counts did not display a Poisson distribution.

Whether or not the fibre distribution on the filter is consistent with the Poisson distribution may depend on the state of aggregation of the fibres being sampled. If a dilute dispersion of single fibres is collected over a long period of time, the distribution on the filter should theoretically conform to the Poisson distribution. If, however, the same number of fibres are collected in a short sampling time from a highly aggregated dispersion, the distribution on the filter will deviate from the Poisson distribution, and the extent of the deviation observed will be strongly influenced by the fibre counting criteria which

TABLE 1. INCIDENCE OF NON-POISSONIAN FIBRE DEPOSITS ON AIR SAMPLE FILTERS

Filter Loading		Airborne Fibre Concentration ** Fibres/mL	Number of Filters Examined		Proportion of Filters on which Fibre Distribution was not Poissonian Percent
Fibres per Field of View*	Fibres/mm <sup>2</sup>		Poissonian Distribution	Non-Poissonian Distribution	
<0.49	<169	<0.60	33	0	0
0.50 - 1.9	170 - 655	0.61 - 2.3	12	8	40
2.0 - 3.4	656 - 1172	2.4 - 4.2	11	6	35
3.5 - 4.9	1173 - 1690	4.3 - 6.0	4	4	50
≥5.0	≥1700	>6.0	4	9	70
TOTAL	-	-	64	27	30

\*Field of View area 0.0029 mm<sup>2</sup>

\*\*Calculated on the basis of a 2 hour sampling period at a flowrate of 2 L/minute, using filter of area 855 mm<sup>2</sup>



are adopted. Thus, deviation of counting data from Poisson behaviour is probably not simply a function of filter loading, but a consequence of the combined effects of aggregation state, fibre concentration, sampling time and arbitrary counting criteria. There is no adequate base of suitable data to examine this problem completely, but it is evident that the Poisson assumption is incorrect for a significant proportion of the samples. Accordingly, it is necessary to establish a conservative method by which the confidence intervals can be specified for all samples.

For a sample in which the Poisson assumption is shown to describe the distribution adequately, the total number of fibres counted defines the 95% confidence interval, which can be obtained from published tables<sup>37</sup>. Under these conditions the 95% confidence interval is independent of the number of fields of view examined. Although the Poisson distribution is asymmetrical about its peak, it becomes more symmetrical as the number of fibres counted is increased. For fibre counts exceeding about 30 fibres, the Poisson distribution can be approximated by a Gaussian having the same mean, but with a variance equal to the mean. The 95% confidence interval can then be computed using the Student's "t" distribution. The Gaussian approximation approaches the Poisson distribution more precisely as the number of fibres counted is increased, and above 100 fibres the difference between the two distributions is negligible.

When a sufficient number of fibres has been counted, and the data are shown not to conform to the Poisson assumption, the distribution is generally broader, and can be described by a Gaussian distribution in which the mean and variance are independent of each other. An estimate of the variance must then be obtained from the fibre counts made on individual fields of view. This can only be accomplished if the total fibre count exceeds about 30.

In the examination of the 91 filters, it was found that the greatest divergences from the Poisson distribution occurred in two samples having loadings of 1840 fibres/mm<sup>2</sup> and 4850 fibres/mm<sup>2</sup> respectively. The corresponding 95% confidence intervals were factors of 1.86 and 1.98 larger than those which would have been calculated on the basis of the Poisson assumption. On this rather limited evidence it is likely that 95% confidence intervals larger than twice that predicted by the Poisson assumption will rarely be exceeded. Figure 1 shows a graphical representation of the deviations from Poisson behaviour observed in the study. It can be seen that:

- (a) non-Poisson behaviour is more serious for high fibre loadings;
- (b) below the normal maximum fibre density of 634 fibres/mm<sup>2</sup> specified in the AIA method, the 95% confidence intervals are not likely to exceed 1.5 times those predicted on the basis of the Poisson distribution; and
- (c) the maximum fibre density of 1667 fibres/mm<sup>2</sup> for the stated optimum range in the USPHS/NIOSH method is sufficiently high that 95% confidence intervals 1.8 times those predicted on the basis of the Poisson distribution may occur. Moreover, in Table 1 it can be seen that non-Poisson fibre counts were obtained on 10 out of 25 filters with fibre densities within the range 656 - 1690 fibres/mm<sup>2</sup>.

It is clear that at high fibre densities, fibre counts (although not necessarily the overall fibre deposit on the filter) frequently depart from Poissonian behaviour. Accordingly, it is recommended that the 95% confidence interval should be calculated using Gaussian statistics in those cases where departures from the Poisson distribution are demonstrated. The frequency with which non-Poisson fibre counts are obtained can also be reduced by imposing a lower value for the maximum recommended fibre density.

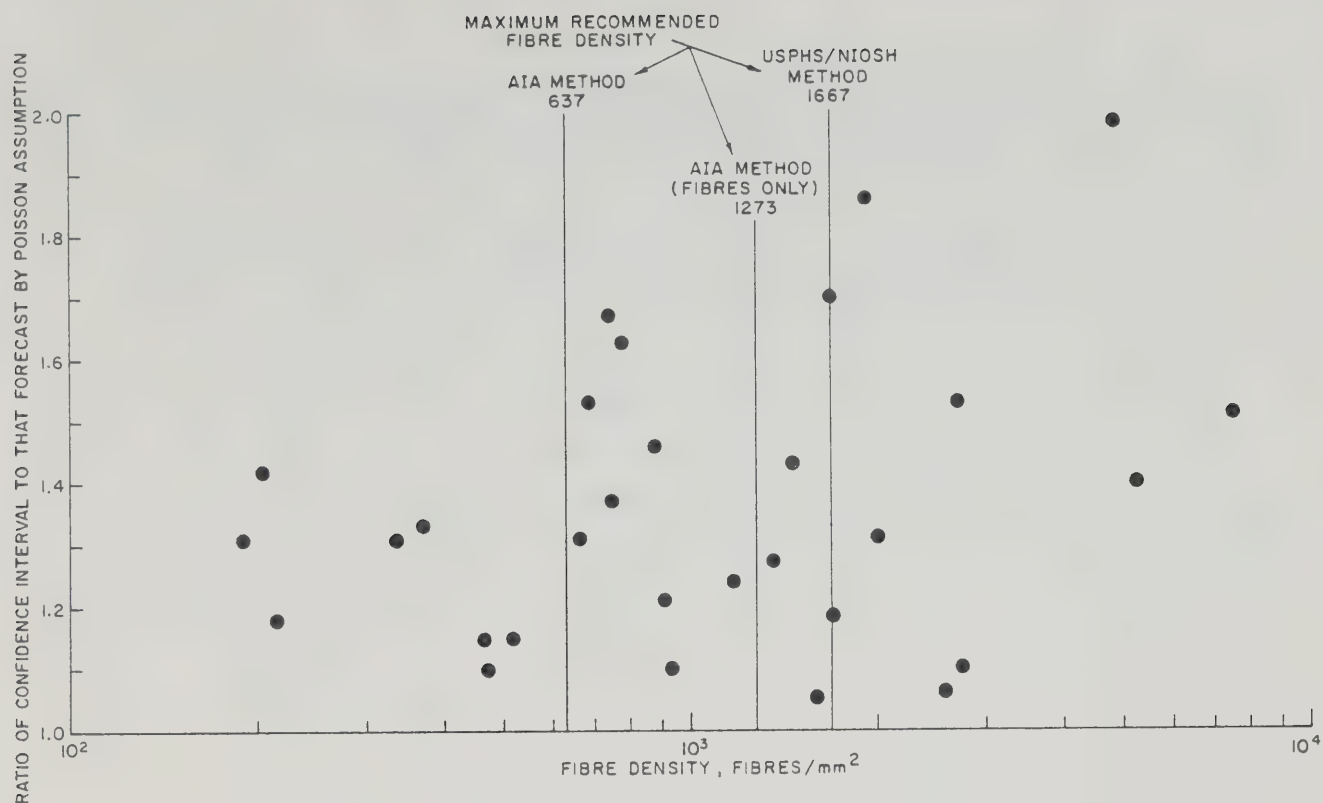


Figure 1. Deviation of Field of View Fibre Counts from the Poisson Distribution.

It is instructive to examine the consequences of the Poisson assumption, and the observed deviations from it, on the precision of a single measurement. It is desirable to express the precision in terms of a 95% confidence interval, which is the range of values within which 95% of repeat measurements should fall. Table 2 shows the 95% confidence limits for the Poisson distribution. The values in parentheses represent the corresponding limits within which 95% of the non-Poisson samples should fall, based on the experimental observations. Although below about 30 fibres it is not possible to calculate meaningful standard deviations from the individual field of view fibre counts, non-Poissonian behaviour can sometimes be demonstrated for low fibre counts. In these cases the analyses should either be rejected, or a confidence interval could be assumed which is less optimistic than that predicted by the Poisson distribution.

TABLE 2. 95% CONFIDENCE LIMITS FOR THE POISSON DISTRIBUTION  
AND OBSERVED DEVIATIONS FOR HIGH FIBRE DENSITIES

Observed Number of Fibres	Lower Limit Number of Fibres	Upper Limit Number of Fibres
0	0	3.7
1	0.03	5.6
2	0.24	7.2
3	0.62	8.8
4	1.1	10.2
5	1.6	11.7
6	2.2	13.1
7	2.8	14.4
8	3.5	15.8
9	4.1	17.1
10	4.8	18.4
15	8.4	24.7
20	12.2	30.9
25	16.2	36.9
30	20.2 (13.1)	42.8 (47.0)
35	24.4 (16.8)	48.7 (53.2)
40	28.6 (20.6)	54.5 (59.4)
45	32.8 (24.4)	60.2 (65.6)
50	37.1 (28.4)	65.9 (71.6)
100	81.4 (70.0)	122 (130)
200	174 (158)	230 (242)

Values in parentheses represent 95% confidence intervals based on 1.5 times that predicted by the Poisson distribution, symmetrically disposed about the mean. Calculation of these deviations is not possible for less than about 30 fibres.



The detection limit must be carefully discriminated from the analytical sensitivity, which is that concentration which corresponds to the counting of one fibre. The detection limit of the method can be defined as the minimum fibre concentration which can be detected above the background fibre count. If the background fibre count were always zero, the detection limit would be equal to that concentration which can, at 95% confidence, be discriminated from the zero fibre count. Referring to Table 2, the upper 95% confidence limit, even for a fibre count of zero, is 3.7 fibres. If the use of more complex statistical tests is to be avoided, the lower 95% confidence limit of any measurement must exceed 3.7 fibres if the measurement is to be considered detectably higher. In Table 2, it can be found that for this case a mean value of 9 fibres has a lower 95% confidence limit of 4.1 fibres, and can be considered detectable above a zero background.

The detection limit of the method can theoretically be reduced indefinitely by filtration of greater volumes of air. This, however, is not the case. The detection limit is affected by the overall dust loading on the filter, and by artifacts of the clearing process which may appear as "fibres". As the fibre concentration decreases, increase of the volume of air filtered causes more general dust to be collected and the fibre count obtained has little meaning. It is generally agreed<sup>8,9</sup> that the useful lower limit of the membrane filter method is usually between 0.1 and 0.5 fibre/mL. In the USPHS/NIOSH method the lower limit of reliable quantitation is specified as 0.1 fibre/mL, assuming detection of 10 fibres in 100 fields of view and appropriate sampling conditions. In view of the stated acceptable background level of 5 fibres/100 fields of view on unused filters, it is questionable whether 10 fibres/100 fields of view can be regarded as "reliable quantitation." The Poisson 95% confidence intervals for 5 and 10 fibres respectively are 1.6 - 11.7 fibres, and 4.8 - 18.4 fibres. Although a statistical test would show that, at 5% significance, a measurement of 10 fibres could theoretically be shown to be higher than a background represented by

5 fibres, the observed inter-laboratory variability (Section 2.4.3) would usually preclude such a definite statement. Accordingly, 0.1 fibre/mL cannot in any practical sense be considered as the limit of reliable quantitation. This value should be considered only as an approximate detection limit.

There is also a "fibre density effect," which leads to higher estimates of the fibre concentrations from filters on which the fibre densities are low. This is possibly due to the tendency of an operator to examine the sample more closely when there are few fibres present. The same subjective effect is probably responsible for the reporting of higher fibre counts when more time is taken for the examination.

#### 2.4.3 Inter-Laboratory Variability of Fibre Counts

In Section 2.4.2 the considerations of variability were confined only to the theoretical consequences of sampling from a Poisson distribution. This would be the situation if an ideal sample were counted by a single experienced operator who did not display any random or systematic errors. Unfortunately, such operators do not exist, and subjective errors have been found to be the most significant in phase contrast fibre counting. When filters are distributed to different laboratories, and even to different operators within the same laboratory, the fibre count results from a single sample display considerable variability. Crawford and Jones<sup>38</sup> have examined the variability, and have concluded that unambiguous definition of the analytical method is not sufficient to ensure satisfactory reproducibility between counts performed by different counters and laboratories. Crawford<sup>39</sup>, in a submission to the working group developing the ISO/DIS method,

summarized the variations associated with subjectivity as follows:

- (a) between-counter variation, same sample: 10 fold;
- (b) between-counter systematic variation: 2 - 3 fold;
- (c) within-counter random and systematic drift: 2 fold.

Such large variability factors dominate the statistical considerations of Section 2.4.2, and have very serious consequences for the demonstration of compliance.

The extent of the problem is illustrated by Table 3, in which some of the results of an inter-laboratory study conducted by ASTM are presented. For one sample, it was found that the results spread over a range of more than a factor of 6. This indicates that *for the same sample*, subjective factors account for one operator being able to find six times more fibres than another operator.

TABLE 3. INTER-LABORATORY FIBRE COUNTING OF FOUR FILTERS  
USING USPHS/NIOSH METHOD  
(Fibre counts made by 18 experienced counters)

Airborne Fibre Concentration, Fibres/mL (Assuming 4 hour Sampling at 2 Litres/Minute)				
	Asbestos Operation			
	Carding	Twisting	Milling	Milling
Mean Value	1.36	1.00	0.99	0.64
Lowest Value	0.63	0.51	0.33	0.41
Highest Value	3.04	1.43	2.04	1.32
Range (High/Low)	4.8	2.8	6.2	3.2

(Derived from ASTM Committee E34 Unpublished Data)

It has been the practice to use the coefficient of variation as a measure of the variability of inter-laboratory data. The coefficient of variation, also referred to as the relative standard deviation, is the standard deviation divided by the mean value. For a Gaussian distribution, 68% of the measurements lie within a range of plus or minus one standard deviation from the mean value. The coefficient of variation becomes inappropriate for description of the variability of measurements derived from low fibre counts, or if the distribution of the measurements is shown for other reasons not to be Gaussian.

Leidel et al<sup>6</sup> have reviewed the precision of the USPHS/NIOSH method, and Busch et al<sup>41</sup> have examined fibre counts from a series of filters each counted by Johns-Manville personnel. Busch et al concluded that the coefficients of variation were not much greater than the theoretical minimum values predicted by assumption of the Poisson distribution. Chase<sup>42</sup> has commented that the Busch et al analysis did not consider inter-laboratory variability, and that when this was considered, the values of the coefficient of variation were much larger. Moreover, with increase in the number of fibres counted, the observed coefficient of variation did not fall as rapidly as that predicted by the Poisson distribution or by the Busch et al analysis. These data are shown in Figure 2, and it can be seen that Chase's inter-laboratory data indicate a coefficient of variation of about 0.65 for a count of 100 fibres, rising to about 1.0 for counts of less than about 20 fibres. These data seem to be consistent with a published study by Rajhans and Bragg,<sup>43</sup> in which a coefficient of variation of about 0.6 was obtained for concentration measurements in the vicinity of 1 fibre/mL. Unfortunately, the Rajhans and Bragg publication does not indicate the fibre loadings of the filters or the number of fibres counted for each result, and thus the data cannot be easily compared with the work of other authors.



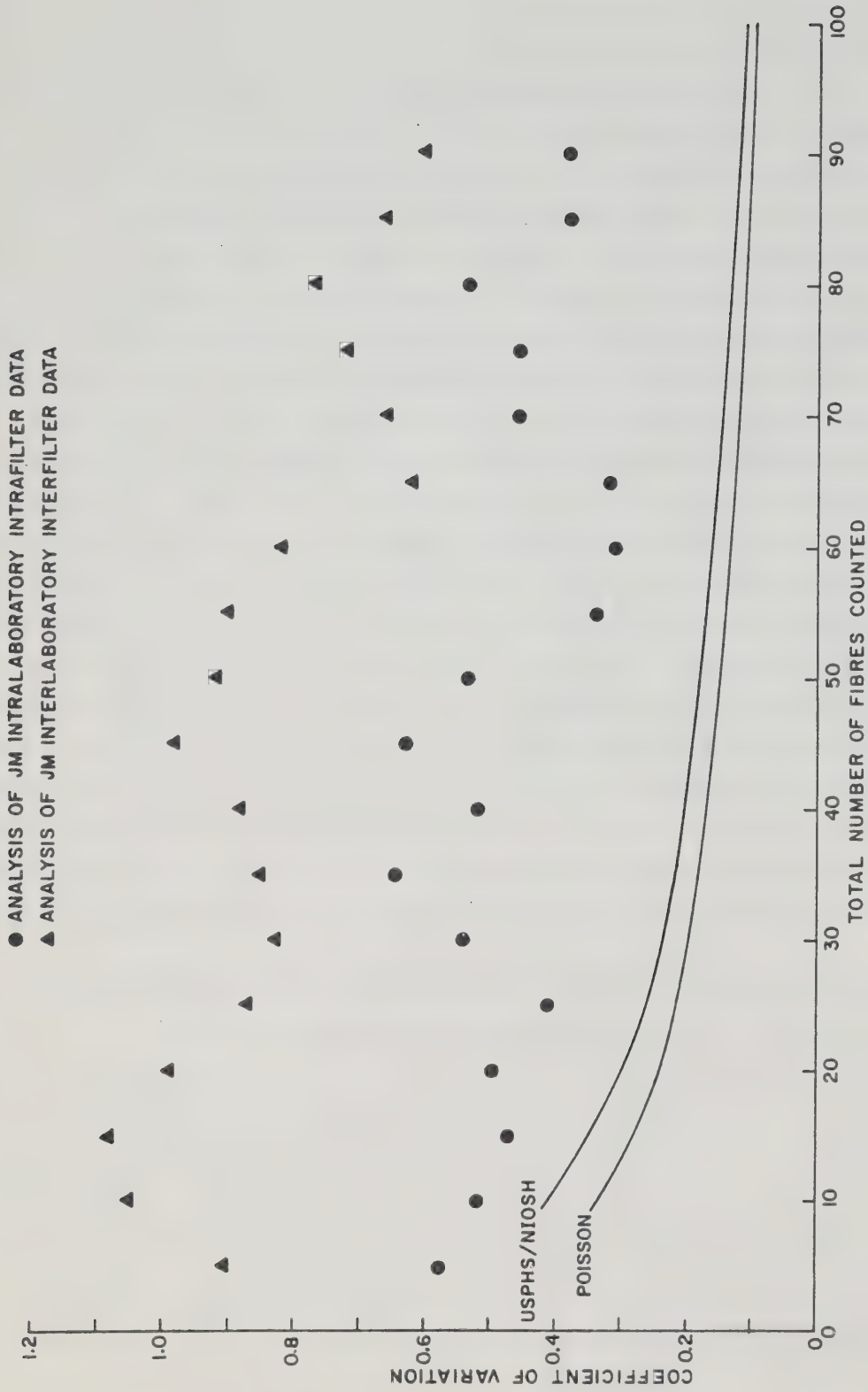


Figure 2. Effect of the Number of Fibres Counted on the Coefficient of Variation for Fibre Counts using the USPHS/NIOSH Membrane Method. (After Chase, 1980).

The study sponsored by Canada and the CEC<sup>23</sup> to establish the optimum fibre counting criteria has not yet been fully evaluated, but some preliminary conclusions can be drawn. In this study, a series of 8 slides, prepared from filters originating from textile, asbestos-cement, brake lining, and amosite operations, were counted by two operators in each of 14 laboratories. The coefficients of variation obtained are shown in Table 4. One laboratory reported results consistently higher than the rest of the group, particularly when the AIA counting criteria were in use, and it is for this reason that the coefficients of variation have been reported both with and without the inclusion of data from this laboratory. In the evaluation of variability of an analytical method, it is of questionable validity to incorporate data from a laboratory which is known *in advance* to produce results consistently a factor of 2 or 3 times higher than those from the rest of the group. Accordingly, the precise interpretation of these data will be somewhat controversial. The coefficients of variation showed some differences when different counting criteria were used. The values reported for the AIA counting criteria are relatively consistent with the work by Chase<sup>42</sup>, and indicate the very large variabilities involved even when the investigation was as well-controlled as the

TABLE 4. CANADA-CEC INTER-LABORATORY STUDY OF FIBRE COUNTING CRITERIA (1982)

COEFFICIENTS OF VARIATION

	Inter-Laboratory Coefficients of Variation		
	AIA Counting Criteria	Modified Version of CRS Criteria; 3:1 Aspect Ratio	Modified Version of CRS Criteria; 5:1 Aspect Ratio
All Data Included	0.25 - 0.85	0.21 - 0.47	0.20 - 0.35
Excluding Data from one Outlier Laboratory	0.22 - 0.56	0.18 - 0.32	0.19 - 0.30

Canada-CEC study. Use of the modified CRS fibre counting criteria appeared to reduce the observed variability, but it is also clear that the variability depended on the type of sample. The range of fibre densities was such that no fewer than about 70 fibres would have been counted for any sample. The coefficients of variation in Table 4 thus correspond to relatively ideal conditions, and would be significantly higher for samples in which close to the minimum number of about 15 fibres are counted. However, compliance with control limits is stated in terms of a single personal sample, and coefficients of variation do not indicate the overall range of actual results obtained. Table 5 shows the maximum and minimum fibre densities reported by the 14 laboratories, for both the AIA and the modified CRS criteria with 5:1 aspect ratio. The latter counting criteria yielded the lowest coefficients of variation, and the results are quoted for comparison. It should also be considered that the result from each laboratory is the mean of counts from two observers, and therefore the actual ranges of values are somewhat larger than those quoted. It is clear that using the AIA criteria, the values reported may occur in a range exceeding an overall factor of 2.1 - 8, depending on the type of sample and who counted it. For the counting criteria

TABLE 5. CANADA-CEC INTER-LABORATORY STUDY OF FIBRE COUNTING CRITERIA (1982)  
REPORTED FIBRE DENSITIES

Fibre Counting Criteria		Reported Fibre Densities, Fibres/mm <sup>2</sup>							
		Amosite	Brake Lining		Textile		Asbestos-Cement		
		1	1	2	1	2	1	2	3
AIA Method	High Value	690	560	330	560	770	720	850	1290
	Low Value	330	110	120	160	300	90	160	170
Modified CRS Criteria 5:1 Aspect Ratio	High Value	660	300	300	390	840	320	430	950
	Low Value	240	150	140	130	370	120	210	280

which displayed the least inter-laboratory variability, the corresponding range was a factor of 2.0 - 3.4. It appears from these results that counting of asbestos-cement plant samples gave rise to more variability than for the other types of sample; particularly when using the AIA counting criteria.

The Ontario Ministry of Labour regularly monitors the airborne fibre levels in a number of Ontario industries. As part of the October 1980 survey in the Mississauga plant of Certified Brakes Ltd., sample filters collected by both the Ontario Ministry of Labour and Certified Brakes Ltd. were counted by operators from both the company and the government laboratories. The results obtained are shown in Table 6, and in graphical form in Figure 3. This is an example of fibre counting by two laboratories that, judging by the considerable amount of sampling performed by the Ontario Ministry of Labour, have had substantial experience in collaborative counting of samples of the same character from a single plant. Accordingly, the agreement between the values at high fibre concentrations is excellent. However, at concentrations below 1 fibre/mL the agreement is poor. Samples reported as 0.14 fibre/mL and 1.0 fibre/mL by Certified Brakes Ltd. are both reported as 0.3 fibre/mL by the MOL Laboratory. Although the study contains few results in this region below 1 fibre/mL, they illustrate the difficulty of obtaining results sufficiently reliable to administer control limits of 0.2 fibre/mL and 0.5 fibre/mL on the basis of a single sample.

#### 2.4.4 Summary of Inter-Laboratory Measurements

There is an extensive amount of published material concerning the precision of the membrane filter method, much of which cannot be compared in detail because of differences between individual methods



TABLE 6. INTER-LABORATORY COMPARISON OF FIBRE COUNTING  
RESULTS BY CERTIFIED BRAKES LTD. AND THE ONTARIO MINISTRY OF LABOUR

Samples Collected By:	Sample Number	Fibre Count (Fibres/mL)	
		Certified Brakes Ltd.	Ontario Ministry of Labour
Ontario Ministry of Labour	12	3.7	4.3
	5	3.8	2.2
	22	8.7	7.3
	30	1.2	0.9
	24	1.0	1.2
	38	1.3	1.3
	35	19.0	13.0
Certified Brakes Ltd.	3-1	0.14	0.3
	2-1	8.3	9.3
	2-2	0.27	0.38
	3-2	20.0	22.0
	4-1	1.0	0.3

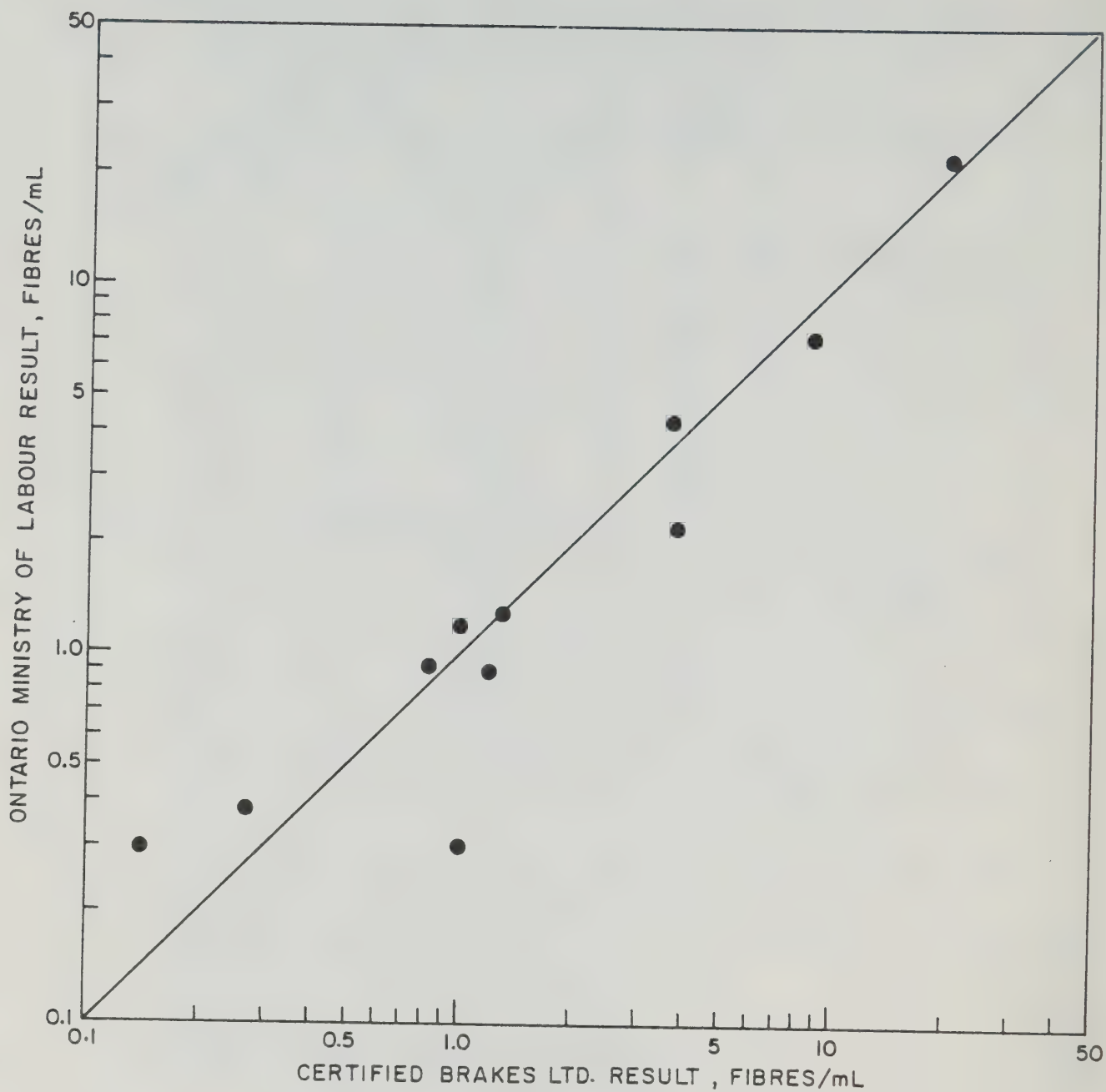


Figure 3. Inter-Laboratory Comparison of Fibre Counting by Two Laboratories with Experience in Collaborative Counting.

and the samples used for comparison. For example, as mentioned in Section 2.3.1, the relative fibre counts made by Teichert<sup>29</sup>, using the acetone-Triacetin and the acetone-Euparal mounting methods, are not necessarily in conflict with the values obtained by Ogden<sup>30</sup>. The samples used for the two studies were not identical, and a difference in the width distributions could explain the apparently contradictory results. In the same way, the coefficients of variation quoted in some studies cannot be fully evaluated, because reference has not been made either to the actual form of the distribution or to the numbers of fibres counted for each result. When only low numbers of fibres have been counted, or when widely-dispersed inter-laboratory data are involved, assumption of Gaussian statistics is not always justified, and the coefficients of variation derived may not adequately describe the dispersion of the data.

Systematic variations of a factor of 3 have been observed between individual laboratories that were using analysis procedures apparently identical in all respects. This is exemplified by the fact that two groups which individually display good intra-laboratory agreement can quite consistently produce counts which differ by a factor of 3 or more. In counting trials between a number of laboratories in the United Kingdom and laboratories in Sweden, the values reported from the Swedish laboratories were between 2 and 3 times higher than those of the U.K. laboratories.<sup>44</sup> In each country, a central laboratory operates a programme of inter-laboratory exchanges of samples, and *all* laboratories performing fibre counting participate in these exchanges.

It can be concluded that subjectivity remains the most significant factor which causes variability in membrane filter counting and that coefficients of variation are unlikely to be lower than 0.3 - 0.5, even under well-controlled conditions. These coefficients of variation refer to sample evaluation only; a further variability component will arise from the air sampling procedure. There appears to be no published data on the overall variability, starting with several parallel samples collected separately on the same worker.

The need for a continuous programme of training and sample exchange is demonstrated by the results of the inter-laboratory studies, and in particular by the systematic discrepancy between the United Kingdom and Sweden. It appears that the only way in which agreement between laboratories can be achieved is by continuous sample exchange. Therefore, it is recommended that a Central Reference System be established. Operation of such a system by an independent agency with experience in asbestos measurement, and participation by both government and industry, would establish the credibility of routine data.

## 2.5 Demonstration of Compliance or Non-Compliance with Legislated Control Limits

Control limits, or threshold limit values (TLV), are established by regulatory bodies and are maximum airborne concentrations above which some corrective action should be taken. The law may require that no measurement of an airborne concentration exceed a legislated fixed value, and thus any measured value which does exceed it is considered to be out of compliance. The Ontario Regulations, and also those of other regulatory agencies, are specified in this manner. The difficulty of applying this procedure, particularly if it is taken to the point of prosecution, is illustrated by Figure 4. The curve shows the frequency distribution of replicate concentration measurements about their mean value. The mean value in this case



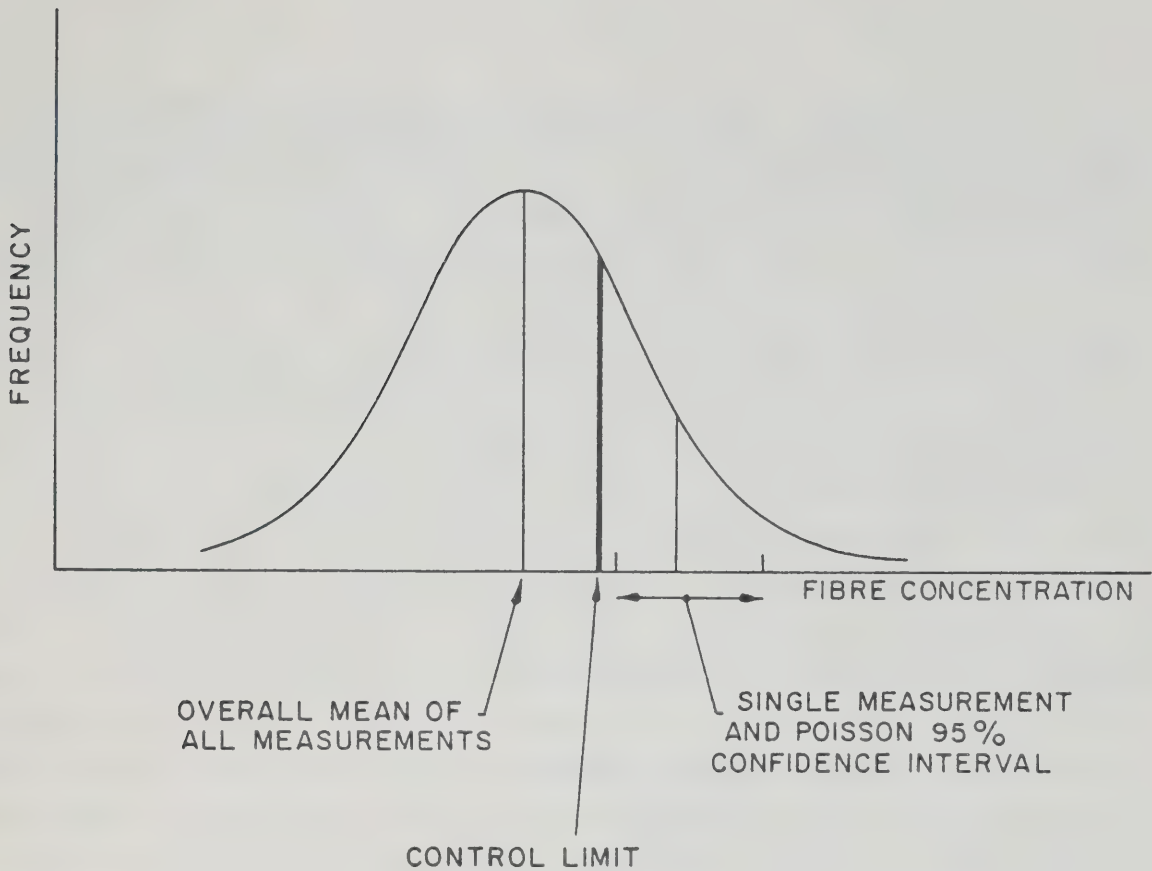


Figure 4. Illustration of the Relationship Between the Overall Mean, a Single Measurement, and a Control Limit.

is shown as lower than the control limit. A single measurement could frequently occur which has a sample estimate of the mean higher than the control limit. In this illustration, Poisson errors alone for the single sample result would indicate that even its lower 95% confidence limit exceeded the control limit. On the basis of the single sample, therefore, the measurement is out of compliance, even though the true concentration, represented by the overall mean, would be in compliance. A prosecution based on this single measurement, therefore, could obviously be challenged on the grounds that the high value was a consequence of measurement error.

This argument is usually of minor importance in determination of compliance, since the measurements of most controlled substances can be made with coefficients of variation less than 0.1.<sup>45</sup> Asbestos fibre counting is a major exception in that the coefficients of variation for the measurement are very large, often exceeding 0.6, and therefore, these must be taken into account in compliance determination. Moreover, at very low fibre concentrations, the observed inter-laboratory variability is such that the distribution of replicate measurements is skewed and Gaussian statistics are not adequate to describe the measurements. It is clear that in the determination of compliance with control limits, all the very large errors associated with asbestos fibre counting must be accommodated. Accordingly, in order to demonstrate compliance, an industry should be required to show that at some acceptable level of confidence all of the individual personal sample measurements are below the relevant control limit. In a similar way, a regulatory body which wishes to demonstrate non-compliance would be required to show, at an acceptable level of confidence, that a particular value is above the control limit. The level of confidence selected depends on what is considered an acceptable risk that a particular result may be outside the confidence interval. For industry, this means that in order to comply with a particular control limit, the mean airborne fibre concentration to which each worker is exposed must be very much lower than the specified limit if this limit is to be exceeded only rarely. For the regulatory body wishing to demonstrate non-compliance with a control limit, the high value in question must be sufficiently above the limit that there is only a small risk that it was a consequence of measurement error.

The subjective and statistical errors associated with the membrane filter technique used for monitoring of asbestos fibre concentrations have been shown to be very large. Consequently, the industry must maintain average airborne concentrations correspondingly lower in order to accommodate these measurement errors, and a demonstration of non-compliance requires observation

of a value significantly above the control limit. In view of the variability of the membrane filter method, it is unlikely that measurements above the control limit will never be observed, even in situations where the true concentration never exceeds the control limit.

A number of factors enter into the possibility of demonstration of compliance or non-compliance:

- (a) the actual variability of the worksite air concentrations;
- (b) the variability associated with counting of samples;
- (c) the analytical detection limit;
- (d) the relationship of the control limit to the analytical detection limit;
- (e) the maximum acceptable sampling period;
- (f) the proportion of the legislated time-weighted average (TWA) period for which samples were collected; and
- (g) the acceptable risk that the mean concentration is not contained within the calculated confidence interval.

Item (f) in the above list is particularly relevant to the Ontario Regulation in which a 40-hour TWA is specified. In the United Kingdom it was recognized that "for both operational and enforcement reasons it is not easily practicable to set limits related to periods of more than about 4 hours."<sup>46</sup> Presumably, the 8-hour period was selected in the USA by the Occupational Safety and Health Administration<sup>47</sup> for similar reasons. It is clear that for practical reasons, the 40-hour TWA, specified in the Ontario Regulation, will not be simple to enforce. In the Ministry of Labour records,

many instances can already be found where workers have refused to carry the personal samplers, even for the limited periods and relatively infrequent sampling studies so far conducted. From the point of view of technical effort alone, it seems unacceptable to sample each employee continuously for one week; an operation which according to current Ministry records would result in between 10 and 30 samples per employee *for a single determination*. Alternatively, if short period samples are taken at intervals during the 40 hours, the calculation of the TWA contains supposition by the technician as to the level and continuity of exposure during the intervening periods when samples were not being collected. This supposition would be challenged in any prosecution, and therefore such calculations have limited value. Leidel et al<sup>45</sup> recommend that the sampled period should cover at least 70% to 80% of the full specified period, which is not difficult to achieve for an 8-hour TWA. Moreover, from the point of view of compliance, they also suggest that the employer should assign the exposure average of the sampled period to the whole period, and that the compliance officer should assume no exposure during the period when samples were not being collected. It is clear, therefore, that for enforcement of compliance with a 40-hour TWA, sampling for 28 - 32 hours would be necessary, with an assumption of zero exposure for the remaining 8 - 12 hours. The technical effort required per employee is still formidable, and on past evidence it is questionable if employees would co-operate by wearing personal samplers for the required period of time.

It is recommended that the arguments for the 40-hour TWA be reviewed in the light of the U.K. and U.S. regulations, and in consideration of the level of technical and analytical effort required for enforcement.



### 2.5.1 Demonstration of Compliance Based on a Single Sample or Time-Weighted Average

For demonstration of compliance or non-compliance, it is usual to consider that a 5% risk of making the wrong decision is acceptable. To demonstrate compliance, the employer requires the upper, one-sided 95% confidence limit of the measurement to be lower than the control limit, and to demonstrate non-compliance, the compliance officer requires the corresponding lower 95% confidence limit to be higher than the control limit. If either of these conditions is met, there is only a 5% probability that the decision is incorrect.

The theoretical limitation imposed by the Poisson distribution, together with the deviations observed for non-Poissonian behaviour at high fibre densities, define the most optimistic situation for demonstration of compliance with a control limit. This assumes that whoever counted the sample is counting at levels appropriate for comparison with the control limit. These theoretical limitations, however, are overshadowed by other limitations which are a consequence of the large inter-laboratory variabilities.

There is no reference standard against which fibre counting measurements can be compared, and the question therefore arises as to how fibre counts can be compared against a control limit. While no reference standard exists, and fibre counts are made by more than one laboratory, the only way in which compliance with a control limit can be determined is by taking account of the *inter*-laboratory variability. This is achieved by calculation of the one-sided 95% confidence limits according to the expressions:

$$(a) \text{ Lower 95\% Confidence Limit (LCL) } = \text{Mean} - 1.645 \times \text{CV} \times \text{Mean}$$

$$(b) \text{ Upper 95\% Confidence Limit (UCL) } = \text{Mean} + 1.645 \times \text{CV} \times \text{Mean}$$

If UCL is lower than the control limit, the employer can be 95% confident of compliance; if LCL exceeds the control limit, the compliance officer can specify non-compliance with 95% confidence. The expressions are of doubtful validity when the value of LCL becomes negative, as is the case when the coefficient of variation is high. It should be noted that these expressions are different from those published in the USPHS/NIOSH method,<sup>6</sup> which deviate from the 5% probability criterion depending on the relative values of the mean and the control limit.

Because the trend has been towards compliance with lower fibre concentration control limits, the effects of background fibre counts on demonstration of compliance must also be considered. All of the membrane filter methods acknowledge that backgrounds of "several countable fibres" per 100 fields of view often exist on unused filters. Unfortunately, the number of background fibres fluctuates from filter to filter, and unless there is a long experience of zero background filters, the presence of background fibres must be assumed. This is relatively unimportant when more than 50 fibres are counted on the actual sample. However, the low control limits for both crocidolite and amosite require interpretation of low fibre counts if realistic sampling periods are to be used. For application of these low control limits, the effects of background fibres are most serious, and must be considered in the interpretation of the fibre counting results. It is also important that an extensive blank control filter programme be operated. In such a programme, blank filters should be randomly mixed with the actual samples, and counted without the counter being aware of their nature. Moreover, if the blank control filters are to be really representative, they should carry non-asbestos particulate deposits at the same level as the actual samples. However, this usually cannot be achieved, and

unused filters are submitted as blanks. The USPHS/NIOSH method actually specifies that these should be labelled as blank filters. It is generally unsound practice for the individual who performs the fibre counts to be aware of which samples are controls or blanks. The blank filters should first be placed at random positions in the series of sample filters, and a random number similar to the sample numbers should then be issued to each filter.

If the counting results do not provide discrimination of the actual samples from the blank filters, the significance of the results must be questioned. In the AIA method, 3 fibres in 100 fields of view is considered to be a representative limiting value for the background. Assuming the Poisson distribution, the one-sided upper 95% confidence limit for 3 fibres is 7.75 fibres, obtained from the published tables.<sup>37</sup> In order to demonstrate clearly that a measurement exceeds the background value, it would be desirable for the lower 95% confidence limit of any measurement to exceed the upper 95% confidence limit of the blank, in this case 7.75 fibres. Any overlap of the confidence limits could introduce controversy regarding the interpretation. Assuming once again that the samples do not depart from a Poisson distribution, the published tables show that a one-sided lower 95% confidence limit of 8.46 fibres corresponds to a mean value of 14 fibres.

Thus, if there is a mean blank value of 3 fibres/100 fields of view, mean counts of less than 14 fibres/100 fields of view should not be considered significant. The Ontario MOL Code for Measurement and the USPHS/NIOSH method are somewhat less discriminating than the AIA method in respect of background acceptance, and about 3 fibres on 50 smaller fields of view are considered acceptable. In this case,

fibre counts lower than 19 fibres/100 fields of view should not be considered significant as a measurement if the 95% confidence intervals of blank and sample distributions are to be completely separated. Using the MOL method, for a 90-minute sample, this would yield a mean fibre concentration of 0.16 fibre/mL, with a 95% confidence interval of 0.10 - 0.25 fibre/mL. Thus there is a situation in which the stated acceptable background fibre count defines the minimum sample fibre count which can be reliably discriminated from the background, and this minimum fibre count has a 95% confidence interval which contains the proposed control limit for crocidolite. This argument is based solely on Poisson statistics, and if the inter-laboratory data of Section 2.4.3 are taken into account, the situation is much worse, depending upon the values assumed for the coefficient of variation.

A study has been made of the data collected by the Occupational Health Branch of the Ontario Ministry of Labour. It appears that for operational reasons a sampling period of 90 minutes was selected for almost all samples. The minimum significant fibre count above the background has been shown to correspond to 0.16 fibre/mL for a 90-minute sample. Table 7 shows the effect on determination of compliance if a minimum fibre count above background and various measured values of the coefficient of variation are assumed. In the case of the Canada-CEC study data, the largest coefficients of variation were selected. Use of the optimum counting rules reduced these to smaller values between 0.19 and 0.35, depending on the type of sample. It is important to recognize that the values obtained in this study correspond to samples in which a minimum of 70 fibres were counted, and that for



TABLE 7. EFFECTS OF SELECTED COEFFICIENTS OF VARIATION ON  
MINIMUM DETECTABLE CONCENTRATION FOR 90 MINUTE SAMPLES

(Mean Fibre Concentration = 0.16 fibre/mL)

Origin	Coefficient of Variation	Number of Fibres Counted for Which Coefficient of Variation Applies	95% Confidence Limits, Fibre/mL	
			LCL*	UCL
USPHS/NIOSH Method <sup>6</sup>	0.3	20	0.08	0.24
Canada-CEC <sup>2,3</sup> AIA Fibre Counts, One Laboratory Excluded	0.56	Not Specified, but Exceeding 70	0	0.31
Rajhans and Bragg <sup>4,3</sup>	0.60	Not Specified, but corresponding to 1 fibre/mL	0	0.32
Canada-CEC <sup>2,3</sup> AIA Fibre Counts, All Laboratories	0.85	Not Specified, but Exceeding 70	(0)	0.38
Chase (Johns-Manville) <sup>4,2</sup>	1.0	20	(0)	0.42
Rendall and Andrew <sup>4,8</sup>	1.5	20	(0)	0.55

\*Values in parentheses were negative and are defaulted to zero.

lower numbers of fibres the coefficients of variation would be significantly higher. It is likely that for counts of 20 fibres a coefficient of variation between about 0.6 and 1.0 can be expected. In Table 7 it is clear that regardless of the value selected for the coefficient of variation, all UCL values exceed 0.2 fibre/mL, and an employer could not therefore demonstrate compliance with a 0.2 fibre/mL control limit. Moreover, the LCL values are either very low, zero or negative, and a compliance officer would not be able to demonstrate non-compliance with the 0.2 fibre/mL control limit. If the Chase data are used, it is questionable whether a 90-minute sample, which gives a UCL of 0.42 fibre/mL, could be relied on as an employer compliance demonstration for a control limit of 0.5 fibre/mL, since Gaussian statistics are not appropriate at low fibre counts.

If the sampling period is extended to the practical maximum of 6 hours, the theoretical lowest significant measurement, above background, would correspond to 0.04 fibre/mL, with a one-sided 95% upper confidence limit of 0.06 fibre/mL to 0.14 fibre/mL, depending on the coefficient of variation used. These calculations are consistent with the detection limit of the AIA method, which is stated to be between 0.1 fibre/mL and 0.5 fibre/mL, regardless of the volume of air filtered.<sup>8</sup>

The effect of the coefficient of variation on the demonstration of compliance or non-compliance is shown by consideration of Table 8. The LCL and UCL values have been calculated for a range of fibre concentrations, assuming several different coefficients of variation. The data from the Canada-CEC study, and those of Rajhans and Bragg, indicate that it would be most unwise to assume that at this time coefficients of variation less than 0.4 are prevalent even for high fibre counts. The actual values may be much higher. For a coefficient of variation of 0.4, Table 8 shows that, using a 6-hour sample, the employer can theoretically

TABLE 8. EFFECT OF INTER-LABORATORY COEFFICIENT OF VARIATION ON COMPLIANCE DEMONSTRATION

Mean Fibre Concentration, Fibres/mL	Number <sup>+</sup> of Fibres Counted		Coefficient of Variation												
			USPHS/NIOSH <sup>++</sup>		0.3		0.4		0.5		0.6		0.8		
6 Hour Sample		90 Minute Sample		LCL	UCL	LCL	UCL	LCL	UCL	LCL	UCL	LCL*	UCL	LCL*	UCL
0.10	48	12	0.07	0.13	0.05	0.15	0.03	0.17	0.02	0.18	0	0.20	0	0.23	
0.16	76	19	0.12	0.20	0.08	0.24	0.05	0.27	0.03	0.28	0	0.32	0	0.36	
0.20	96	24	0.16	0.24	0.13	0.30	0.07	0.33	0.04	0.36	0	0.40	0	0.46	
0.30	100	36	0.24	0.36	0.20	0.45	0.11	0.50	0.06	0.54	0	0.60	0	0.69	
0.50	100	60	0.40	0.60	0.25	0.75	0.15	0.85	0.10	0.90	0	1.0	0	1.2	
0.60	100	72	0.48	0.72	0.30	0.90	0.18	1.0	0.12	1.1	0	1.2	0	1.4	
0.70	100	84	0.56	0.84	0.35	1.1	0.21	1.2	0.14	1.3	0	1.4	0	1.6	
1.0	100	100	0.80	1.2	0.50	1.5	0.30	1.7	0.20	1.8	0	2.0	0	2.3	
1.5	100	100	1.2	1.8	0.75	2.3	0.45	2.6	0.30	2.7	0	3.0	0	3.5	
2.0	100	100	1.6	2.4	1.0	3.0	0.70	3.3	0.40	3.6	0	4.0	0	4.6	
3.0	100	100	2.4	3.6	1.5	4.5	1.1	5.0	0.60	5.4	0	6.0	0	7.0	
5.0	100	100	4.0	6.0	2.5	7.5	1.8	8.3	1.0	9.0	0	10	0	12	

<sup>+</sup>Assuming 8.55 cm<sup>2</sup> Active Area of Filter;  
2 litres per minute sampling rate; and  
0.00585 µm<sup>2</sup> graticule area.

<sup>++</sup>USPHS/NIOSH calculated coefficient of variation based on the total number of fibres counted.  
Values quoted are for the 6 hour sample.

\*Negative Values defaulted to zero.

just achieve a compliance demonstration with a standard of 0.2 fibre/mL, since the UCL value of 0.17 fibre/mL corresponds to a measurement at the detection limit of 0.1 fibre/mL. However, it is not likely that the coefficient of variation will be as low as 0.4 for fibre counts of less than 50 fibres, and more significantly, other dust will usually overload the filter in a shorter period than 6 hours. For the 6-hour sample, and a coefficient of variation of 0.4, the compliance officer would require a measured fibre concentration of 0.7 fibre/mL, yielding an LCL value of 0.21 fibre/mL, before non-compliance could be demonstrated.

For the proposed amosite control limit of 0.5 fibre/mL, the employer and the compliance officer would require mean fibre concentrations of 0.3 fibre/mL and 1.6 fibres/mL to demonstrate compliance or non-compliance respectively. For the proposed chrysotile control limit of 1 fibre/mL, the corresponding values would be 0.6 fibre/mL and 3.0 fibres/mL. This general approach is consistent with that published by Ogden,<sup>49</sup> who applied a rather lower intra-laboratory coefficient of variation of about 0.2 to the demonstration of compliance at 95% confidence.

In Table 8 it can be seen that tests by the compliance officer are not possible for coefficients of variation of 0.6 and greater, for any control limit, since the LCL values are all zero or negative. The employer's compliance test is still possible at the higher coefficients of variation for the proposed amosite and chrysotile control limits of 0.5 fibre/mL and 1.0 fibre/mL, but successful demonstration of compliance requires lower values for the measurements.

At high coefficients of variation, the distribution of measurements is probably not Gaussian, and the values in Table 8 should therefore be used with some caution. No measurements of the variability for samples with low fibre counts appear to be available, and thus the form of the distribution is currently a matter of speculation.



In summary, demonstration of compliance or non-compliance is seriously affected by the coefficient of variation of inter-laboratory fibre counts, and non-compliance for any control limit cannot be proven if the coefficient of variation exceeds about 0.6. The actual value of the coefficient of variation is probably a function of sample type, and certainly becomes larger with reduction in the fibre count. Ogden<sup>49</sup> recommends that more than 50 fibres should be counted for optimum precision with a minimum of effort.

The above discussion indicates that demonstration by the employer of compliance with a control limit of 0.2 fibre/mL, even with a 6-hour sampling period, is probably not possible for a number of practical reasons. For sampling periods less than 6 hours, the employer's compliance demonstration is not possible if the variability of fibre counting observed in recent studies is typical, and if reasonable background fibre counts are assumed.

The calculations in this section have all been based on the coefficients of variation observed when chrysotile fibres have been counted. Unfortunately, no reliable data can be found regarding the variability of crocidolite fibre counts, and because of differences in the fibre width distributions, this material cannot be considered as similar to amosite, for which only limited data are available. Use of data derived from low fibre counts for amphibole asbestos is subject to many uncertainties. These include a serious lack of experimental data for amphibole (crocidolite and amosite) fibre counting reproducibility. It is possible that, in view of the larger refractive index of amphibole fibres, and their generally larger diameters, the reproducibility may approach the theoretical values and be less prone to subjectivity. However, this is not known. Using the available data, it appears that for a single sample, a satisfactory demonstration of compliance or non-compliance with a

0.2 fibre/mL control limit is not possible. This conclusion applies equally to a small series of samples collected during a work shift which are used to compute a time-weighted average, since each sample is subject to the background and variability effects. It is likely that the Ontario 0.5 fibre/mL control limit for amosite will allow a demonstration of compliance, but only under very well-controlled analytical conditions.

It is recommended that research be conducted into the membrane filter measurement of fibre concentrations below 1 fibre/mL for chrysotile, 0.5 fibre/mL for amosite and 0.2 fibre/mL for crocidolite, to determine the reliability of such fibre counts. It is also recommended that single sample fibre counts demonstrating compliance or non-compliance with the amosite and crocidolite control limits be treated with caution.

#### 2.5.2 Demonstration of Long Term Compliance

When a large number of samples are taken in a plant which uses asbestos, the fibre counts obtained are found to be distributed according to a logarithmic-normal distribution. In this distribution the logarithms of the fibre counts are distributed normally, and then conventional Gaussian statistics can be used. Accordingly, the appropriate parameters to describe the distribution of the fibre counts are the geometric mean and the geometric standard deviation. For a logarithmic-normal distribution the geometric mean and the median have the same value, but the arithmetic mean is higher than the median. It should be appreciated that for a logarithmic-normal distribution, the arithmetic mean is largely controlled by the few highest values, whereas the geometric mean and median are not weighted in this way. The meaning of the various parameters when used to specify average

fibre concentrations should be carefully understood. The distribution will contain a wide range of concentration measurements. A proportion of these will exceed the control limit, either because the concentration was actually higher than the limit, or because a few high values have occurred as a result of measurement error. In order to demonstrate that only a small proportion of the fibre concentration measurements exceed the control limit, the median fibre concentration must be maintained very much lower than the control limit. The extent to which it is lower is controlled by the overall variability of the fibre counts obtained, expressed as the geometric standard deviation (GSD).

For specified compliance levels, the factors between the long-term median fibre concentration and the control limit can be obtained by calculation from the logarithmic-normal distribution. These are shown in Table 9.

TABLE 9. RELATIONSHIP BETWEEN A CONTROL LIMIT AND THE LONG-TERM AVERAGE OF LOGARITHMIC-NORMALLY DISTRIBUTED SINGLE SAMPLES (AFTER HSE 1979)<sup>50</sup>

Geometric Standard Deviation of the Individual Sample Distribution	FACTOR BY WHICH CONTROL LIMIT EXCEEDS LONG-TERM AVERAGE		
	Proportion of Samples Which Comply with Control Limit		
	95%	99%	99.9%
1.5	1.80	2.37	3.22
2.0	2.47	3.95	6.70
3.0	3.35	7.07	16.30
4.0	3.77	9.67	27.74

The situation is illustrated by a study of the Ontario Ministry of Labour surveys conducted at the Mississauga plant of Certified Brakes Ltd. The results for 4 separate surveys are shown in Figure 5. It can be seen that the results are distributed approximately logarithmic-normally, and if the latest data from January 1981 are considered, 95% of the results were below 2.4 fibres/mL with a plant median of 0.34 fibre/mL. The concept of the plant average, of course, only holds true if the mix of occupations selected for sampling is representative. Moreover, improvements can only be monitored by repeat measurements distributed in a similar manner between the various work stations. As can be seen in Table 9, the fibre concentration below which the median must be maintained in order to demonstrate compliance by

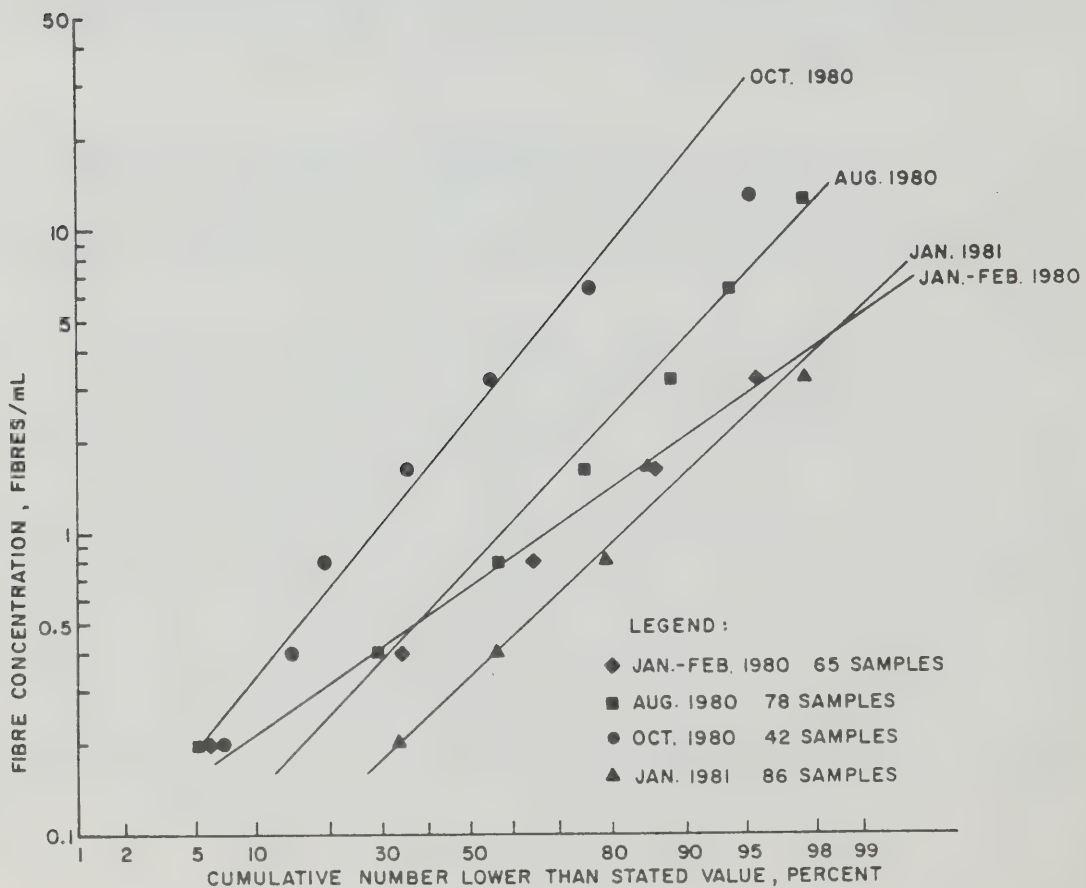


Figure 5. Results of 4 Separate Surveys by the Ontario Ministry of Labour to Measure Airborne Fibre Levels at the Mississauga Plant of Certified Brakes Ltd.



a fixed proportion of the samples depends on the GSD of the distribution. The observed GSD is higher than that of the real fibre concentrations, because of the contribution by the variability of the fibre counting method, thus making a demonstration of compliance more difficult. If the January 1981 survey is taken to be representative of the combination of fibre concentration and counting variabilities, the GSD of 3.2 can be used to predict some aspects of the measurement problem. Figure 6 shows the control limits of 1.0, 0.5 and 0.2 fibre/mL plotted at the 95% compliance level. The lines through these points all represent a GSD of 3.2. The detection level of the measurement is taken to be 0.1 fibre/mL. For crocidolite, demonstration of 95% compliance requires that 86% of the fibre counts are below the detection level of 0.1 fibre/mL. From the previous considerations, this is not considered possible in any real industrial situation. For amosite, the requirement is for 61% of the fibre counts to be below the detection level of 0.1 fibre/mL. It is considered that this is

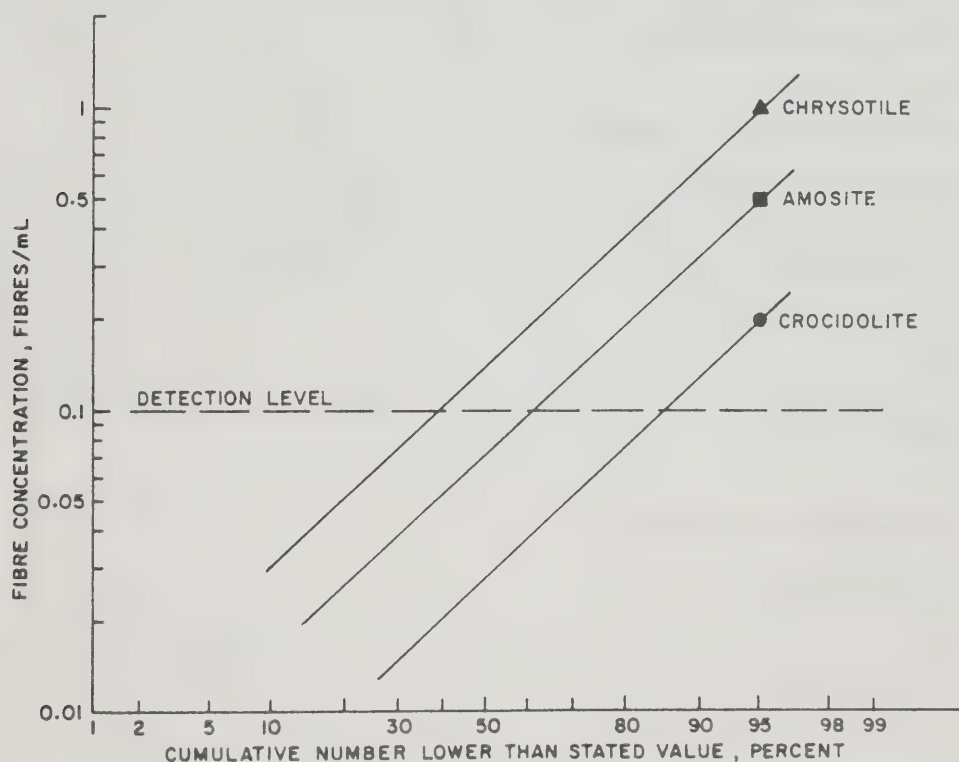


Figure 6. Distributions of Single Sample Results Required to Demonstrate Compliance at the Three Ontario Control Limits, Assuming a Geometric Standard Deviation of 3.2.

possible where asbestos forms the majority of the airborne dust, but whether it can be achieved generally will probably depend on the actual proportion of asbestos. For chrysotile, compliance requires that 40% of the fibre counts are below 0.1 fibre/mL. This is also considered possible if a well-controlled analytical quality assurance programme is instituted.

It is important to recognize the effect of analytical variability on the demonstration of long-term compliance, since the GSD values from the Certified Brakes Ltd. data are substantially higher than the range of 1.5 to 1.8 used in the submission by Chase and Rhodes<sup>51</sup>, based on work by Leidel et al<sup>45</sup>. If the Certified Brakes plant is representative of those studied by Leidel et al, it would suggest that a very substantial component of analytical variability was present in the MOL data from which Figure 5 was derived. A consequence of such analytical variability would be that a larger proportion of the measurements would be stated to exceed the control limit than actually was the case.

### 3. IDENTIFICATION OF FIBRE TYPE AND SIZE

In operations where mixed asbestos varieties are in use, it would be desirable for the fibre concentrations of each variety to be reported separately, since a different value of control limit is proposed for each variety. Moreover, in view of the works of Stanton<sup>52</sup> and Pott,<sup>53</sup> it would also be desirable to separate the fibre counts into different dimensional ranges. These ranges would be <5  $\mu\text{m}$ , between 5  $\mu\text{m}$  and 10  $\mu\text{m}$ , and >10  $\mu\text{m}$ .

#### 3.1 Fibre Identification Methods

##### 3.1.1 Optical Methods

Conventional polarized light microscopy (PLM) and dispersion staining optical microscopy (DSM) are both convenient ways to identify fibres in optical microscope samples. However, neither technique is

useful for samples which are mounted in a medium of fixed refractive index. To employ either technique, there is a requirement for examination of the sample in a range of refractive index media, and this cannot easily be accomplished with mounted membrane filter samples. The technique by LeGuen et al,<sup>25</sup> in which the filter is collapsed by acetone vapour and the fibres exposed by slightly etching the filter in a low temperature asher, may offer a method which can be applied. However, the optical identification techniques are not feasible on a routine basis for fibres less than 1 - 2  $\mu\text{m}$  in diameter, although the DSM technique has been claimed to be useful down to 0.3  $\mu\text{m}$ .<sup>54</sup>

If the primary objective is merely to separate chrysotile from amphiboles, it would perhaps be possible to prepare two slides, one of which would contain a filter sector mounted in a refractive index medium closely matching that of chrysotile. In this case, only the amphibole count could be recorded, since the chrysotile fibres would display very little contrast. The second slide would provide the total fibre count. Use of this technique carries the assumption that chrysotile and amphibole are the only fibrous species present. The method would require a substantial development effort, but implementation afterwards would be simple. However, since two samples would be counted, the cost of analysis would be double the current cost.

### 3.1.2 Electron Optical Methods

The scanning electron microscope (SEM) with energy dispersive X-ray analysis (EDXA) is the most appropriate and inexpensive means for identification of individual small fibres in workplace atmospheres. In contrast to the situation in the environment, the minerals which may be present are known, and therefore identification can be based on a

simple observation of the X-ray spectrum. Fibres down to about 0.05  $\mu\text{m}$  diameter can be detected on a modern SEM, and analyses can be obtained for fibres of this diameter if they are well separated from other particles or fibres.<sup>55</sup> This restriction is a consequence of the fact that the X-ray resolution is only about 1  $\mu\text{m}$ . Discrimination between the major asbestos varieties is rapid, since acquisition of an X-ray spectrum need go no further than to identify the major peaks. Nevertheless, the technique is a more expensive one than the phase contrast method. It has been used by Ontario Research Foundation in cases where gypsum was present along with amosite in insulation removal monitoring.

### 3.1.3 Other Instrumental Methods

Some discrimination between chrysotile and the amphiboles can be made on the basis of the different ways in which fibres of these materials align in magnetic fields. This technique was pioneered by Timbrell,<sup>20</sup> and recently an instrument based on this principle has become commercially available.<sup>14</sup> The instrumental aspects of this technique have been refined,<sup>56</sup> and current research in this area is discussed in Section 4.1.3.

The use of X-ray diffraction for fibre identification has been suggested. However, when other dusts are present, interference from these can be a significant problem. The discrimination of chrysotile from non-fibrous serpentine minerals is particularly difficult.<sup>57</sup> A method for measurement of chrysotile or amphibole in the presence of interfering non-fibrous minerals has been developed, which has a detection limit of 0.1 microgram ( $\mu\text{g}$ ).<sup>58</sup>



Infra-red spectroscopy has been found to be a useful identification method for sub-milligram amounts of single varieties of asbestos.<sup>59</sup> However, the technique is subject to interferences from other minerals and is therefore not considered useful for analysis of asbestos mixed with other dusts.<sup>60</sup>

### 3.2 Fibre Dimension Measurement

Although the Asbestosis Research Council phase contrast method originally specified the option of sizing the fibres into 5 size ranges, it is clear that the reproducibility would be degraded even further if an additional task of this type were placed on the microscopist. Moreover, the information produced would be of marginal value, since only fibres down to 0.2  $\mu\text{m}$  diameter would be seen. The Pott carcinogenicity hypothesis<sup>53</sup> yields a series of curves which peak at about 0.1 - 0.2  $\mu\text{m}$  diameter, and if this hypothesis is accepted, many biologically-relevant fibres are not recorded. In contrast, the SEM is capable of detecting fibres throughout most of the envelope of Pott's curves. If individual fibre dimensions are required, the SEM offers the only realistic instrumental approach. The sample preparation is direct, and substantially simpler than that required for the phase contrast optical method. The statement that measurements made on an SEM cannot be related to the optical fibre counts now used<sup>51</sup> is not correct. This has simply not yet been attempted. Before the considerable advantages of the SEM are discounted, it should perhaps be phased in along with the optical method for comparison studies. More development work is required to assess its performance in measurement of fibre dimensions, but there is every reason to expect it to be superior to that of the membrane filter method, particularly for the size range of fibres currently counted.

#### 4. FUTURE METHOD DEVELOPMENTS

It is clear that the membrane filter method remains the least expensive technique for personal monitoring of asbestos exposure. In spite of its limitations, it has given good service in monitoring fibre concentrations in the asbestos industry. However, the studies made of its variability show that there are still serious limitations which become more significant as attempts are made to apply it to measurement of lower fibre concentrations. There are a number of new approaches to the problem of fibre measurement which are under development.

##### 4.1 Automated Phase Contrast Optical Fibre Counting

An image analysis device (Magiscan)<sup>10</sup> has been applied to the problem of phase contrast fibre counting. Software has been developed which allows counting of constituent fibres of aggregates to a constant set of criteria. The use of the automated technique eliminates the subjectivity of the manual fibre counter, but introduces other problems, not the least of which is expense. The equipment cost is approximately \$200,000, at 1982 prices. The Health and Safety Executive (HSE) laboratory in the U.K. is developing entirely automated slide changing and focusing techniques. Much development is required before the instrument can be used routinely and automatically. There are also problems of logic<sup>61</sup> and of spurious fibre counts originating from the residual granularity of the mounting medium.<sup>27</sup> Moreover, in view of the fact that operators focus the microscope through some depth of the filter when counting by the membrane methods, it is not clear whether the Magiscan result represents the same features as those which would be counted manually. It has been found that the Magiscan generally produces higher counts than the mean of those obtained by manual fibre counting, although the results are within the overall range of manual results.

#### 4.2 Automated SEM Fibre Counting and Identification

The SEM-EDXA system has many advantages over the optical phase contrast microscope, some of which have already been discussed. A computer has been interfaced with such an instrument, and the combination has been applied to the fibre counting problem.<sup>11,56</sup> The high depth of focus of the SEM is a considerable advantage since once the instrument has been focused on a sample, there is no need to adjust it again for minor sample height differences. The back-scattered electron image mode, using modern detectors, allows even small fibres to be imaged with good contrast. The automated instrument counts fibres, including the constituent fibres of aggregates, according to constant criteria, and then reports a length, width, and location within the field of view, for each. The fibres are then automatically and individually identified on the basis of a 10 - 20 second count of the X-ray emission, if this information is required. Although currently at a feasibility demonstration stage, the instrument has shown that the fibre counting logic is satisfactory, and that a system can be assembled with very little custom-made electronic equipment. Sample changing equipment could be very simple in design. The overall cost would be about \$250,000, at 1982 prices. The time for analysis could be as little as 10 minutes per sample for fibre sizing and counting, with an additional 20 minutes for EDXA analyses, if these are required.

#### 4.3 Magnetic Alignment with Scattered Light Measurement

The work by Timbrell<sup>20</sup> has been applied in a new instrument manufactured by Vickers Instruments.<sup>14</sup> Essentially, the membrane filter is dissolved in solvent on a microscope slide which is held between the poles of a magnet. The fibres adopt some alignment relative to the magnetic field, and are then permanently fixed when the solvent has

evaporated. The light scattered by the aligned fibre sample is then measured as a function of angle. The instrument must be calibrated against manual fibre counts for each size distribution encountered. The detection level is stated to be about 0.1 fibre/mL, the limitation probably being a function of granularity of the filter.

The aligned fibre technique has been investigated in two versions by Chatfield and Riis,<sup>62,63</sup> and direct examination of fibre suspensions in liquids has been found to overcome the limitations associated with the filter. For this technique, the fibres can be removed from the filter, and suspended in water, after which a magnetic field is rotated around a spectrophotometer cell containing the suspension. The fibres rotate with the magnetic field, and measurements of scattered light are made. The measurement time is only a few minutes. The technique is reproducible, even for samples of concentrations below 0.1 fibre/mL, and it appears that a detection level of about 0.001 fibre/mL will be possible. The measurement, however, is not currently easy to interpret in terms of numerical fibre concentration. Nevertheless, it is likely that research now in progress will permit fibre dimensions to be obtained, and direct determination of numerical fibre concentrations in samples with different and unknown size distributions would then be possible. Well-characterized reference samples can also be introduced as standards. It is possible to discriminate some of the amphiboles from chrysotile, and it may be possible to obtain separate values for the concentration of different varieties of asbestos in mixed exposure situations. The equipment cost would currently be about \$30,000 (1982 prices).



#### 4.4 Electrostatic Alignment and X-ray Diffraction

It has been found possible to align fibres electrostatically, and the X-ray diffraction pattern approximates that from a single fibre. The method pioneered in the U.S.A. by Birks et al<sup>58</sup> was found to have a detection level of about 0.1 microgram ( $\mu\text{g}$ ) of chrysotile. The detection limit on real samples where interferences are present has not yet been evaluated. The method is simple in principle, but the specimen preparation effort required may make the technique unattractive. Moreover, fibre dimension information cannot be obtained.

#### 4.5 Automated TEM Fibre Counting and Identification

The objection to the use of the SEM on the basis of the resolution limitation can be overcome by using the TEM in a scanning mode. The equipment costs are high (\$500,000), and there are no immediate prospects for automated sample exchange or operation. In conventional TEM mode, the image could readily be examined by an image analysis device, but automation of fibre identification is currently a remote development possibility.

### 5. CONCLUSIONS AND RECOMMENDATIONS

#### 5.1 Membrane Filter Methods

The membrane filter method has serious deficiencies for monitoring of worker exposure to asbestos, but in view of the number and frequency of measurements required, there is currently no fully-developed alternative method which could be immediately implemented. There are, however, other methods under development which will provide greater reliability of measurement.

The variability of the membrane filter method is very high, and inter-laboratory coefficients of variation between 0.25 and 1.0 have been

observed in various studies. Inter-laboratory coefficients of variation up to 0.85 have been observed for ideally-loaded filters even in well-controlled studies. At the low filter loadings obtained when monitoring fibre concentrations below 1.0 fibre/mL, this variability will be particularly serious and will present major problems of interpretation. There are few sources of reliable intra- and inter-laboratory fibre counting variability data which can be related to low fibre counts, particularly for amphibole asbestos. It is therefore important that the best available techniques for phase contrast fibre counting should be used, in order to minimize the variability. As a result of this review of the available versions of the membrane filter method, a number of recommendations can be made which refer particularly to the code of measurement published by the Ontario Ministry of Labour.

- (a) Ontario should adopt the main features of the AIA Reference Method for analysis of membrane filters, in particular the acetone-Triacetin mounting technique and the Walton/Beckett graticule. The acetone-Triacetin method provides superior contrast. Unlike the MOL method, the acetone-Triacetin method provides a permanent filter mount which can be re-evaluated later, if necessary. The Walton/Beckett graticule standardizes the area of sample examined for each field of view, variation of which has been found to introduce inter-operator variability. In addition, the Walton/Beckett graticule is designed specifically for counting fibres, and therefore its use reduces the subjectivity of counting.
- (b) The Ontario method of analysis should incorporate tests of microscope resolution for asbestos fibres.
- (c) The Ontario method of analysis should incorporate improved analytical quality assurance requirements, particularly in the area of random blank and control samples, and "blind" re-counting by a second operator.

- (d) The variability of fibre counting is controlled by the fibre counting criteria adopted. It is recommended that the AIA counting criteria should be incorporated in the Ontario MOL method. It is also recommended that these be replaced by the ISO/DIS counting criteria when international agreement has been reached at the working group level.

A major recommendation concerns the current lack of control over inter-laboratory variability in Ontario, and in Canada as a whole. It is recommended that a Central Reference System be established, which for credibility reasons should be operated by an independent (non-industry, non-government) laboratory with experience in asbestos measurement. The central reference system operated in the United Kingdom is an example. Before being authorized to perform fibre counts, all laboratories should then be required to participate in routine sample exchanges on a continuous basis in order to maintain proficiency. Operation of the system by an independent agency, with participation by both government and industry, would establish the credibility of routine data.

## 5.2 Determination of Compliance with Control Limits

The personal air sampler currently offers the best method of obtaining samples which are representative of a worker's exposure. However, it is clear that for enforcement reasons the total sampling period must be a major proportion of the time over which a time-weighted average is calculated. Accordingly, it appears that to enforce a 40-hour TWA, 10 - 30 samples would be required per worker for each determination, assuming that the worker could be persuaded to wear the sampler for the majority of a working week. It is therefore recommended that the Ontario 40-hour TWA be reviewed as to the level of technical and analytical effort required for enforcement, particularly in the light of the U.K. and U.S. regulations.

The errors of single sample results are very large, and a significant proportion of the filters do not conform to a Poisson distribution. It is

recommended that 95% confidence intervals be reported, along with a test for the Poisson distribution, for each sample in which compliance or non-compliance with control limits is to be established. Compliance determination must take account of the measurement uncertainties, and compliance criteria should be stated in the conclusions derived from the results.

Using the current membrane filter method, compliance with a 0.2 fibre/mL control limit cannot be unequivocally demonstrated since analytical variability estimates are widely dispersed, even for a sampling period of 6 hours. For a 90-minute sampling period, a compliance demonstration is not currently possible. It is possible that the 0.5 fibre/mL level may also be subject to similar difficulties. Compliance with the 1 fibre/mL control limit can be demonstrated if satisfactory analytical quality assurance techniques are in use. More research is needed to establish methods for the control of analytical variability at low fibre counting levels. Accordingly, it is recommended that the reliability of fibre counting at concentrations below 1.0 fibre/mL should be established by a logically designed experimental programme. This programme should include chrysotile, amosite and crocidolite.

The arithmetic mean, when used to express average long-term concentration levels, is strongly weighted by a minority of high concentration measurements. It is recommended that long-term average levels be determined using the logarithmic-normal distribution, since the data have been shown to conform to this distribution. However, long-term average levels cannot be determined accurately for either crocidolite or amosite if 95% of the results are in compliance with the Ontario control limits, since this would demand that a large proportion of the measurements be made below the detection level.



### 5.3 Measurement of Exposure to Mixed Varieties of Asbestos

The current membrane filter method is unsuitable for evaluating exposure to mixed asbestos varieties, except as a total fibre concentration measurement. The scanning electron microscope is the only realistic, currently-available approach to separate reporting of fibre counts for the principal asbestos types. In mixed fibre situations, it is recommended that some SEM measurements be conducted in parallel with the routine membrane filter measurements. It is possible that the membrane filter method could be modified so that it would provide the current total fibre count and a separate fibre count for amphibole fibres. This would double the cost of analysis since two samples would be counted. It is recommended that more studies be made of the analytical problem posed by use of mixed asbestos varieties, and asbestos mixed with other species such as gypsum.

### 5.4 Extension of Fibre Concentration Measurements to Include Fibre Lengths

It is not considered practical to attempt sizing of the fibres during a phase contrast fibre count. The method already contains an appreciable subjectivity factor, and the additional load on the operator would significantly increase the variability of the results. For comparison with the Stanton and Pott studies,<sup>52,53</sup> fibre widths are also important, and here the SEM and TEM are currently the only instruments by which these can be obtained. No results of parallel SEM-optical fibre counting determinations have yet been published, and it is recommended that a study of this type be made in a range of industrial locations. There is every reason to expect the SEM performance to be superior to that of the phase contrast optical method.

### 5.5 Improved Fibre Measurement Methods Under Current Development

Automated fibre counting systems on both the optical microscope and the SEM, and systems based on magnetic alignment with light scattering, offer the most promising measurement systems for the immediate future. Other measurement methods do not have the potential for determination of fibre dimensions, which should be a priority requirement for any analytical technique intended to supersede the membrane filter method.

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